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## Urine as Biobased Fertilizer: The Netherlands as Case Study

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### Abstract:

Humankind has grown ever more dependent on mineral fertilizer to feed a growing world population. However, nutrients are rarely recovered and recycled after consumption, leading to environmental waste issues now and potential supply issues in the future. Urine is rich in these nutrients and therefore a prime candidate as precursor for biobased fertilizers. This work presents a case study to remove contaminants – predominantly pharmaceuticals – from human urine using activated carbon adsorbents in an effort to adhere to Dutch fertilizer legislation. In the urine, three pharmaceuticals were identified by target analysis and 11 other contaminants by suspect screening using liquid chromatography quadrupole time of flight mass spectrometry (LC-QTOF/MS). These were successfully removed by treatments with both granulated and powdered activated carbon, albeit with a loss of 16-17% and 2-4% of dissolved nutrients, respectively. Further screening of inorganic contaminants and persistent organic pollutants by inductively coupled plasma mass spectrometry (ICP-MS) and gas chromatography quadrupole-orbitrap mass spectrometry (GC-Q-Orbitrap/MS) showed that all prerequisites for fertilizer status are met, paving the way for its future legal use as biobased fertilizer in the Netherlands.

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# Urine as Biobased Fertilizer: The Netherlands as Case Study




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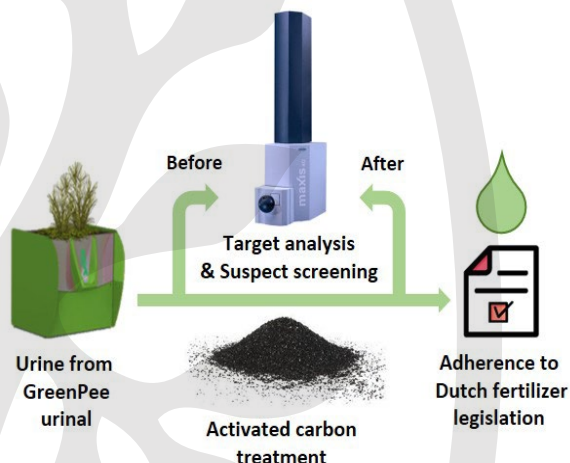
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## SIGNIFICANCE (max 590 characters)

- Activated carbon (AC) adsorbents are able to remove practically all detected contaminants from urine – notably pharmaceuticals – in a fast and facile way
- Both target analysis and suspect screening were performed in this study to allow a thorough assessment of potentially present substances of concern
- This study shows AC-treated urine adheres to all prerequisites listed in the Dutch fertilizer legislative framework for a material to be considered as fertilizer



## Keywords

Nutrient recycling, biobased fertilizer, activated carbon adsorbent, pharmaceutical removal, fertilizer legislative framework, target analysis, suspect screening

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## ABSTRACT

Humankind has grown ever more dependent on mineral fertilizer to feed a growing world population. However, nutrients are rarely recovered and recycled after consumption, leading to environmental waste issues now and potential supply issues in the future. Urine is rich in these nutrients and therefore a prime candidate as precursor for biobased fertilizers. This work presents a case study to remove potentially present contaminants from human urine using activated carbon adsorbents in an effort to adhere to Dutch fertilizer legislation. In the urine, three pharmaceuticals were identified by target analysis and 11 other contaminants by suspect screening using liquid chromatography quadrupole time of flight mass spectrometry (LC-QTOF/MS). These were successfully removed by treatments with both granulated and powdered activated carbon, albeit with a loss of 16-17% and 2-4% of dissolved nutrients, respectively. Further screening of inorganic contaminants and persistent organic pollutants by inductively coupled plasma mass spectrometry (ICP-MS) and gas chromatography quadrupole-orbitrap mass spectrometry (GC-Q-Orbitrap/MS) showed that all prerequisites for fertilizer status are met, paving the way for its future legal use as biobased fertilizer in the Netherlands.

## Introduction

Phosphorus (P), nitrogen (N), and potassium (K) are vital macronutrients for life.<sup>1</sup> Fertilization is essential for high crop yields, historically achieved through the recycling of nutrient-rich organic matter sources such as manure.<sup>2</sup> However, the 19<sup>th</sup>-century discovery of phosphate rock (PR) and the Haber-Bosch process for nitrogen fixation in the following century revolutionized agriculture.<sup>3,4</sup> Today, mineral and chemical fertilizers are indispensable, supporting about 50% of food production.<sup>4-6</sup> However, PR is a finite resource, with depletion projected between 50 to 400 years from now.<sup>7,8</sup> Deposits are unevenly distributed globally, with 75% located in Morocco and the Western Sahara.<sup>8</sup> Furthermore, PR often contains persistent heavy metal (HM) contaminants, such as cadmium (Cd) and uranium (U), which can pose serious health risks.<sup>9,10</sup> As mined PR quality declines, this issue will worsen.<sup>11</sup> Although Haber-Bosch N fixation has no contamination problem, it is energy-intensive, consuming 1-2% of the world's energy production and is responsible for about 1.8% of global CO<sub>2</sub> emissions.<sup>12,13</sup>

Both P and N are used inefficiently. In 2019, 89% of the 21 million tonnes (Mt) mined P was intended for fertilizer production, yet only about 16% of mined P – or 14% of fertilizer P – was actually consumed by people due to agricultural runoff and losses along the agri-food chain.<sup>7,14-17</sup> N efficiency is similar, with 16% of the 100 Mt N fixed annually for fertilization being consumed by humans.<sup>12</sup> Following consumption, nutrients enter wastewater (WW) in areas with sanitary infrastructure. With increasing population growth, urbanization, and sewage system development, P and N flows in WW are expected to rise significantly.<sup>18</sup> As such, WW nutrient recovery and recycling are essential to move away from current linear practices.<sup>19-21</sup> Currently, however, less than 10% of P and generally even less N is recovered from WW – 3% in the case of Paris, for example.<sup>7,22</sup> This contributes to environmental issues like eutrophication, harming marine ecosystems, and perturbing biogeochemical cycles as underlined by the Planetary Boundaries framework.<sup>23-25</sup>

Urban WW's primary nutrient contributor is urine, accounting for <1% of the volume but contributing 80% N, 70% K, and 50% P.<sup>26,27</sup> Approximately 85% of N in urine is excreted as urea. This is rapidly hydrolyzed to ammonia due to the presence of urease, elevating the pH to about 9.<sup>27-29</sup> Both ammonia and phosphate can be recovered, primarily by means of inorganic salt precipitation such as struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6 \text{H}_2\text{O}$ ), K-struvite ( $\text{MgKPO}_4 \cdot 6 \text{H}_2\text{O}$ ), or calcium phosphates ( $\text{CaHPO}_4 \cdot 2 \text{H}_2\text{O}$  /  $\text{Ca}_3(\text{PO}_4)_2$ ).<sup>20,30</sup> However, these products have low solubility thus releasing nutrients slowly, and often requiring further acid treatment to produce more soluble fertilizers.<sup>31</sup> Furthermore, precipitation usually involves chemical additives, like magnesium (Mg) or calcium (Ca) sources.<sup>20,29</sup> Alternatively, ammoniacal N can be recovered through ion exchange or air stripping and absorption via acid trapping.<sup>32-34</sup> The downsides are that ion exchangers are often quickly saturated and require frequent regeneration, and air stripping is generally time-consuming and

inefficient for waste streams with high ammonia concentrations.<sup>35</sup>

Furthermore, as with precipitation, acid trapping requires chemical additives to bind the ammonia.<sup>36</sup>

Urine itself can also serve as a fertilizer, but requires stabilization to prevent N loss as volatilized ammonia.<sup>29,30</sup> This can be achieved through nitrification, converting ammonia to nitrate via intermediate nitrite formation using bacteria.<sup>37</sup> Other options include acidification or alkalization to stabilize urea by inhibiting urease below pH 4 or above pH 10.<sup>29,30</sup> A urease inhibitor can also be added, encompassing a variety of compounds binding to the urease enzymatic pocket.<sup>30,38</sup> Post-treatment methods like evaporation, (membrane) distillation, forward/reverse osmosis, or freeze-thawing can reduce urine volume, increasing nutrient concentration or producing solid urine-based fertilizers while improving transportability.<sup>29</sup> Source-separation of urine thus presents an intriguing alternative to end-of-pipe solutions at wastewater treatment plants (WWTPs).<sup>30,39</sup>

Important to consider then is the fact that approximately 64% of pharmaceuticals and/or their metabolites are excreted via urine.<sup>40</sup> Should they end up in the environment they may pose a significant environmental threat as pharmaceuticals are designed to be effective at low concentrations, potentially interfering with biochemical- and physiological processes.<sup>29,30,41</sup> Hence, urine and its derived products must be free of pharmaceuticals for safe use as fertilizing material.<sup>30</sup> Various methods for treating aqueous waste streams for pharmaceutical removal have been explored, including ozonation, UV/peroxidation, nanofiltration, and emerging technologies like photoelectrochemical treatment.<sup>42-46</sup> Each has its advantages and challenges, requiring further research for practical implementation. Another promising method for removing pharmaceuticals is adsorption, which offers advantages such as low energy consumption, cost-effectiveness, ease of operation, and the potential for adsorbent regeneration.<sup>46-49</sup> Various materials have been proposed to this end, including metal-organic frameworks, membranes, and activated carbon (AC).<sup>50</sup> AC – divided into powdered (PAC) and granulated AC (GAC) – is well known for its effectiveness in pharmaceutical adsorption,<sup>51-55</sup> with PAC boasting a larger surface area and GAC being easier to handle.<sup>48,52-54</sup> Furthermore, AC does not induce the formation of pharmaceutical transformation products.<sup>56</sup> It is best used for apolar to moderately polar compound adsorption, however – highly polar compounds ( $\log K_{ow} \geq 0$ ) tend to adsorb less readily,<sup>57</sup> although only few such strongly hydrophilic pharmaceuticals exist. Recent studies have explored the use of PAC and GAC for producing safe fertilizer from (nitrified) urine, yielding promising results.<sup>48,49,55</sup>

However, to penetrate the market and promote urine-based fertilizer recycling, adherence to relevant legislation is vital. In the Netherlands, companies GreenPee<sup>58</sup> and SEMILLA Sanitation<sup>59</sup> started a collaboration aiming to produce liquid fertilizer from source-separated urine.<sup>58,59</sup> Currently, urine is considered as waste rather than a resource, hindering its utilization as a fertilizer.<sup>60</sup> European law permits materials to achieve End-of-Waste (EoW) status under specific conditions, namely having a defined purpose, demand or market, compliance with national technical and

legislative requirements, and posing no environmental or public health risks.<sup>61</sup>

Dutch legislative requirements for fertilizers include sufficient nutrient content and freedom from inorganic and organic contamination. Adhering to these requirements ensures compliance with environmental and health standards.<sup>62,63</sup>

This study focuses on a real-world case of urine recycling as a liquid biobased fertilizer, adhering to Dutch legislative standards. Its primary goal was to remove pharmaceuticals and other micropollutants listed in Dutch fertilizer law from stabilized urine through AC treatment, enabling its legal use as fertilizer in the Netherlands. The removal of pharmaceutical contaminants guided treatment optimization. Subsequently, the treated urine was further investigated for persistent pollutants through target and suspect screening. To the best of our knowledge, no examples in literature exist where a legislative framework is used to assess a urine-based fertilizer's market suitability, although an exception is found in Switzerland, where the urine-based fertilizer Aurin – produced by Vuna – has successfully entered the market.<sup>64</sup> Furthermore, there is only little precedence on urine suspect screening and none combined with contaminant removal.<sup>65</sup>

#### Methods and Materials Dutch Fertilizer Legislation

To facilitate the use of urine as a biobased fertilizer, this study uses the prerequisites for the application for fertilizer status in the Netherlands as framework for conducted treatments and analyses. These can be divided into fertilizing components, inorganic contaminant concentrations, and organic contaminant concentrations, specified here for a liquid organic material.<sup>62</sup> Requirements for fertilizing components are 0.5% wt. of either N, P<sub>2</sub>O<sub>5</sub> or K<sub>2</sub>O in the respective dry matter of the liquid material. Should N be the only fertilizing component, at least 85% has to be bound organically. Alternatively, 20% of dry matter should be organic matter.<sup>62</sup>

Inorganic contaminants refer to heavy metals (HMs) or metalloids. HMs subject to legal limits are cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), nickel (Ni), lead (Pb), and zinc (Zn), as well as arsenic (As). Maximum allowable concentrations are given in Table 1 and are expressed as mg HM per kg of the major fertilizing component, which is defined as the one that would reach either 80 kg P<sub>2</sub>O<sub>5</sub>, 100 kg N, 150 kg K<sub>2</sub>O or 3000 kg of organic matter first with increasing application.<sup>62</sup> In the case of urine, this is N.<sup>28</sup>

Table 1. Maximum inorganic contaminant concentrations allowed for a Dutch fertilizer.<sup>60</sup>

Contaminant	Maximum concentration allowed per fertilizing component		
	(mg kg N <sup>-1</sup> )	(mg kg P <sub>2</sub> O <sub>5</sub> <sup>-1</sup> )	(mg kg K <sub>2</sub> O <sup>-1</sup> )
Cd	25	31.3	16.7
Cr	1500	1875	1000
Cu	1500	1875	1000
Hg	15	18.8	10
Ni	600	750	400
Pb	2000	2500	333
Zn	6000	7500	4000
As	300	375	200

Table 2. Maximum organic contaminant concentrations allowed for a Dutch fertilizer.<sup>60</sup>

Contaminant	Maximum concentration allowed per fertilizing component		
	(mg kg N <sup>-1</sup> )	(mg kg P <sub>2</sub> O <sub>5</sub> <sup>-1</sup> )	(mg kg K <sub>2</sub> O <sup>-1</sup> )
Σ PCDD/PCDF	0.015	0.019	0.010
α-HCH	248	310	165
β-HCH	9.6	12	6.4
γ-HCH (lindane)	0.96	1.2	0.64
HCB	31.2	31	20.8
Aldrin	5.6	7	3.7
Dieldrin	5.6	7	3.7
Σ Aldrin/Dieldrin	5.6	7	3.7
Endrin	5.6	7	3.7
Isodrin	5.6	7	3.7
Σ Endrin/Isodrin	5.6	7	3.7
Σ DDT + DDD + DDE	18.6	23	12.3
PCB-28	14.8	18.5	9.9
PCB-52	14.8	18.5	9.9
PCB-101	60	75	40
PCB-118	60	75	40
PCB-138	60	75	40
PCB-153	60	75	40
PCB-180	60	75	40
Σ 6-PCB (excl. PCB-118)	300	375	200
Naphtalene	480	600	320
Fenanthrene	600	750	400
Anthracene	480	600	320
Fluoranthene	148	185	98
Benzo(a)anthracene	184	230	123
Chrysene	184	230	123
Benzo(k)fluoranthene	216	270	144
Benzo(a)pyrene	232	290	155
Benzo(g,h,i)perylene	168	210	112
Indeno(1,2,3-c,d)pyrene	188	235	125
Σ 10-PAK	9200	11500	6133
Mineral oil	748000	935000	498668

Organic contaminants are split into two different categories: contaminants with a defined concentration limit and those without. Contaminants falling in the first category are persistent organic pollutants (POPs), more specifically polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs) and polychlorinated dibenzodioxins / furans (PCDF/PCDD). Maximum allowable concentrations are again expressed as mg POP per kg of the major fertilizing component and are listed in Table 2.

Contaminants for which no legal limits are set may, in principle, be present in fertilizer. However, their application to soil can either be limited by legal soil concentration limits or “indicative environmental risk” (IER),<sup>62</sup> the latter of which can be derived based on contaminant physicochemical characteristics.<sup>66</sup> In the absence of either parameter, presence of the respective contaminant will result in disqualification of the material as fertilizer.<sup>62</sup> For most pharmaceuticals, neither are available.<sup>67,68</sup> In this study, no IER values were derived and complete removal of pharmaceuticals was targeted as recommended in literature.<sup>29,30</sup>

#### Analyte Selection

The selection of analytes assessed in this study was based on commonly used and frequently studied pharmaceuticals (Table 3).<sup>69,70</sup> Analytical grade (>95% purity) reference- and isotope-labeled standards were purchased from either Sigma Aldrich or Acros Organics and are listed in SI Table S1, as are other auxiliary chemicals.

Table 3. Selected pharmaceutical analytes and relevant characteristics.

Analyte	Formula	pK <sub>a</sub>	Log K <sub>ow</sub>	Description
Atenolol	C <sub>14</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub>	9.54 <sup>71</sup>	0.22 <sup>71</sup>	Beta blocker
Hydrochlorothiazide	C <sub>7</sub> H <sub>8</sub> ClN <sub>3</sub> O <sub>4</sub> S <sub>2</sub>	9.96, 8.87 <sup>71</sup>	-0.03 <sup>71</sup>	Diuretic
Diclofenac	C <sub>14</sub> H <sub>11</sub> Cl <sub>2</sub> NO <sub>2</sub>	3.99 <sup>71</sup>	4.51 <sup>71</sup>	NSAID
Sulfamethoxazole	C <sub>10</sub> H <sub>11</sub> N <sub>3</sub> O <sub>3</sub> S	5.72 <sup>72</sup>	0.68 <sup>72</sup>	Antibiotic
N4-Acetylsulfamethoxazole	C <sub>12</sub> H <sub>13</sub> N <sub>3</sub> O <sub>4</sub> S	5.88 <sup>a</sup>	0.86 <sup>a</sup>	Metabolite of SMX
Ibuprofen	C <sub>13</sub> H <sub>18</sub> O <sub>2</sub>	4.42 <sup>71</sup>	4.13 <sup>71</sup>	NSAID
Naproxen	C <sub>14</sub> H <sub>14</sub> O <sub>3</sub>	4.18 <sup>71</sup>	3.24 <sup>71</sup>	NSAID
Trimethoprim	C <sub>14</sub> H <sub>18</sub> N <sub>4</sub> O <sub>3</sub>	7.13 <sup>72</sup>	0.91 <sup>72</sup>	Antibiotic

<sup>a</sup>: Given values are predicted using Chemaxon.<sup>73</sup>

### Urine Sampling and Stabilization

Urine samples were collected in July 2021 at a Green Pee urinal situated in the city center of Amsterdam.<sup>74</sup> To avoid significant urea hydrolysis, the urease inhibitor solution "Fertiflow Add Green" was added to the collection tank of the urinal, consisting of 2,2-dimethyl-1,3-dioxolan-4-ylmethanol (60–64%) and n-butylthiophosphoric triamide (25–27.5%) in water.<sup>75</sup> The urease inhibitor solution was dosed at 5 mL per 100 L urine. Urine samples were stored at 4 °C prior to treatment and subsequent analysis.

### Activated Carbon Treatment

Collected urine samples were subjected to either PAC or GAC treatment. Treatment with PAC (Norit A SUPRA EUR, surface area 1700 m<sup>2</sup> g<sup>-1</sup>, particle size D<sub>50</sub> 20 μm, pH neutral, Norit, Amersfoort, The Netherlands) was batchwise and performed in a 50 mL round bottom flask. PAC doses of 2 and 3 g L<sup>-1</sup> were achieved by addition of 50 and 75 mg PAC to 25 mL urine, respectively. Experiments were stirred at 400 rpm and room temperature. Samples were taken at regular time intervals by syringe, filtered over a 0.45 μm PTFE disposable syringe filter (Whatman) and stored at 4 °C in glass vials until sample preparation. Treatment with GAC (Norit GAC 1240 W, surface area 1100 m<sup>2</sup> g<sup>-1</sup>, particle size D<sub>50</sub> 1.7 mm, pH alkaline, Norit, Amersfoort, The Netherlands) was performed using a glass column (diameter 1 cm) fitted with a frit filter and tap. 300 mL urine was used per experiment, with GAC loadings of 2, 10 and 30 g L<sup>-1</sup> achieved by addition of 0.6, 3 and 9 g GAC, respectively. Column lengths measured at 1.3, 6.7 and 20 cm, respectively. Percolation rate was controlled using the tap and calculated back after the column was finished, rounded to 15 minute increments. Samples were filtered over a 0.45 μm PTFE disposable syringe filter (Whatman) and stored at 4 °C in glass vials until sample preparation. See SI Table S2 for more information on the used ACs and SI Figure S1 for a diagram of the cleaning process.

### Sample Preparation and Analysis

Sample extraction was conducted based on the methods described in Kovalova *et al.* (2012) and Hernández *et al.* (2015), used for target analysis.<sup>76,77</sup>

Both treated and untreated urine matrices were filtered through a 0.45 μm PTFE disposable syringe filter (Whatman) and diluted 100-fold using a mixture of 0.1% formic acid (99%, ULC-MS, Biosolve) in Milli-Q water (Reference A+ System).

An aliquot of 20 mL of the diluted matrix was taken, to which 5 μL of the isotope-labelled standard mix was added. SPE cartridges (Oasis HLB, 60 mg, 3cc, Waters) were preconditioned with 3 mL of methanol (ULC-MS, Biosolve) followed by 3 mL Milli-Q water for LC-qToF analysis, whereas SPE cartridges (Sep-Pak C<sub>18</sub>, 500 mg, 3cc, Waters) were preconditioned with 5 mL of methanol followed by 5 mL Milli-Q water for GC-qOrbitrap analysis. Diluted samples were loaded on the SPE cartridges and percolated at a rate of 1 drop per 2 sec. Upon completion, the cartridges were dried under vacuum for 15 min. Samples were eluted with 3 mL methanol and 5 mL cyclohexane (glass distilled grade, Rathburn) for LC and GC analysis, respectively at a rate of 1 drop per 2 sec. Sample extracts for LC analysis were evaporated to dryness under a gentle nitrogen stream, reconstituted in 1 mL 10:90 methanol:Milli-Q–0.1% formic acid and filtered through a 0.22 μm polypropylene disposable syringe filter (FilterBio). Sample extracts for GC analysis were evaporated under a gentle nitrogen stream until approximately 0.5 mL was left, which were then increased to 1 mL by adding cyclohexane and filtered over a 0.22 μm PTFE disposable syringe filter (FilterBio) using a glass syringe. All samples were prepared in triplicate and a sample blank was prepared for each set of urine samples.

### Target and Suspect Screening Using LC-QToF-MS

A UHPLC system (Nexera, Shimadzu, Den Bosch, The Netherlands) coupled to a Bruker Daltonics maXis 4G high resolution q-ToF/MS upgraded with HD collision cell and equipped with an ESI source (Leiderdorp, The Netherlands) was used for analysis. The instrumental analysis method was based on previously published work by Narain-Ford *et al.* (2022) and Das *et al.* (2023).<sup>78,79</sup> In short, a reversed-phase Kinetex® 1.7 μm Biphenyl 100 Å, LC Column 150 x 2.1 mm (Phenomenex, Utrecht, The Netherlands) column was used to achieve separation. The mobile phases used for this study were ultrapure water with 0.05% acetic acid (mobile phase A) and MeOH (mobile phase B). The total flow rate was 0.3 mL/min. For the analysis, 20 μL of sample was injected for positive and negative ESI mode analysis. The column oven and tray temperature were 40 and 15 °C, respectively. Data-independent MS/MS scans were obtained for target analysis, while data-dependent MS/MS scans were acquired for suspect screening. TASQ software (version 2021, Bruker Daltonics) was used for data processing for the detection and quantification of target analytes. Details of the quantification method can be found elsewhere,<sup>78,79</sup> and the list of qualifier- and qualifier ions for the target analytes can be found in SI Table S3.

The initial step in the suspect screening process involved manually m/z calibrating all raw HRMS data using Bruker DataAnalysis 4.4 software (Bruker Daltonics). The m/z calibration was conducted within a search range of 0.15 m/z, ensuring a calibration error of less than 0.5 ppm. Subsequently, centroiding and converting the data into mzML format were done in ProteoWizard v.3.0.22119.

Further steps were performed in *R*, using the open-source platform *patRoan* (version 2.0.0).<sup>80</sup> First, all features were found and grouped over multiple replicates using the *OpenMS* algorithm.<sup>81</sup> The amount of feature groups was then narrowed down by means of rule-based filtering. For example, a minimum intensity threshold of 10000 was employed. Other settings can be found in SI Figure S2. Componentization was performed using *RAMclustR*<sup>82</sup> (version 1.2.2), and features from positive and negative ionization data were grouped with a 'sets workflow'.<sup>83</sup> The suspect screening itself was the next filter, where remaining feature groups were compared with a predetermined suspect list and kept if they match. For this, the NORMAN priority contaminants list was used, comprising of 976 chemicals of concern including pharmaceuticals, pesticides, preservatives and industrial chemicals.<sup>84</sup> Finally, feature annotation was performed by retrieving respective MS peak lists using *mzR* (version 2.28.0),<sup>85</sup> and matching them to generated formulae and *in silico*-predicted fragmentation data using *GenForm* and *MetFrag* (version 2.4.5),<sup>86,87</sup> respectively. PubChemLite (January 2021) was used as database.<sup>88</sup> Tentatively identified suspects were subsequently ranked according to their confidence level (1-5) based on the proposition by Schymanski *et al.* (2014) and explained in the supporting information (SI Figure S2).<sup>89</sup> Suspects ranked level 4 and lower were discarded and the remainder manually inspected and compared to MS<sup>2</sup> data from the spectral libraries European MassBank and the MassBank of North America.<sup>90,91</sup>

### Target Analysis Using GC-qOrbitrap/MS

Analyses were performed using Thermo Trace 1300 gas chromatograph and a Thermo Exactive Orbitrap mass spectrometer. An Agilent J&W DB5-MS (30m x 0.25mm x 0.25 μm) column was used to achieve the separation. TriPlus RSH autosampler was used to inject 1 μL of extract, using a pulsed splitless injection with an injector temperature of 250 °C. Helium was used as the carrier gas and was set at a constant flow of 1.2 mL min<sup>-1</sup>. The MS scan range was 50-650 *m/z*. Obtained chromatograms were interpreted with the Excalibur software (Version 3.1, Thermo Fischer Scientific Inc.) using the MS Search Program (Version 2.3, Thermo Fischer Scientific Inc.). Peaks in calibration sample chromatograms were matched to their respective analyte using a National Institute of Standards and Technology (NIST) user library containing detailed MS information of the target analytes. Calibration sample chromatograms were subsequently compared to treated urine sample chromatograms to assess the presence of screened analytes. All screened analytes are listed in SI Table S5.

### Inorganic Measurements

The elemental compositions of two untreated urine samples were elucidated by inductively coupled plasma mass spectrometry (ICP-MS) using an Agilent ICP-MS 7800 according to NEN-EN-ISO 17294-2 and conducted by Groen Agro Control (Oss, The Netherlands). Values and standard deviations are listed in Table 7 and are based on the average of the two datasets.

The N and P content of pre- and post-treated samples were measured using a Skalar San++ 5000 segmented flow analyzer equipped with an SA 1074 auto sampler. Samples were diluted 1000-fold prior to analysis. For the determination of N concentration, the samples were mixed with a potassium peroxodisulfate / sodium hydroxide solution and heated to 70 °C. The solution was subsequently mixed with a borax buffer and brought into an UV-digester. After dialysis, the nitrate content was determined by the Griess reaction after reduction of the nitrate to nitrite using a cadmium-copper reductor. The color was measured at 540 nm. For the determination of P concentration, the diluted samples were reacted with ammonium heptamolybdate and potassium antimony(III) oxide tartrate in an acidic medium to form an antimony-phospho-molybdate complex. This complex was reduced to an intensely blue-colored complex by L(+)-ascorbic acid and measured at 880 nm.

### Quality Control

Method recovery experiments were performed in triplicate using the sample preparation method for LC-QToF-MS analysis described previously in *Sample Preparation and Analysis*, where samples were spiked with known concentrations of analytes pre-extraction. Recoveries were determined according to Eq. 1.

$$\text{Recovery (\%)} = \frac{C_{\text{Spiked Sample}} - C_{\text{Unspiked Sample}}}{C_{\text{Added}}} * 100\% \quad (\text{Eq. 1})$$

Matrix effects (ME) were determined using the post-extraction addition method,<sup>92</sup> according to Eq. 2. Positive values indicate ion enhancement, whereas negative values indicate ion suppression.

$$\text{ME (\%)} = \left( \left( \frac{\text{Peak area in matrix}_{\text{post-extraction}}}{\text{Peak area without matrix}} \right) - 1 \right) * 100\% \quad (\text{Eq. 2})$$

Method performance was evaluated based on linearity of the calibration curves, recoveries, limit of detection (LOD), limit of quantification (LOQ) and matrix effect.

## Results and Discussion

### Quality Control

Recoveries were within 70-130% for 75% of the target analytes and correlation coefficients were > 0.99 in all cases. Both atenolol and trimethoprim showed low recoveries. The limits of detection (LOD) ranged from 0.05 to 1.70 ng mL<sup>-1</sup> and limits of quantification (LOQ) from 0.16 to 5.16 ng mL<sup>-1</sup>. These numbers are further detailed in Table 4. Matrix effects indicated signal suppression for all analytes except atenolol and trimethoprim, which were also the ones with low recoveries. Trimethoprim showed an unusually high matrix effect. Matrix effects were corrected for during sample analysis by addition of stable isotope-labelled internal standards pre-extraction, with the exception of hydrochlorothiazide (see SI Table S3 for standards used).

Table 4. Analysis method performance (n=3).

Analyte	Linearity (R <sup>2</sup> )	Calibration range (ng mL <sup>-1</sup> )	LOQ (ng mL <sup>-1</sup> )	LOD (ng mL <sup>-1</sup> )	Recovery (% ± SD)	Matrix effect (%)
Atenolol	0.996	1-25	5.03	1.66	23 ± 1	+16
Hydrochlorothiazide	0.997	0.5-12.5	2.25	0.74	99 ± 3	-60
Diclofenac	0.997	0.2-5	0.92	0.30	90 ± 6	-25
Sulfamethoxazole	0.996	1-25	1.79	0.59	82 ± 7	-44
N4-Acetyl sulfamethoxazole	0.999	0.2-5	0.41	0.14	128 ± 2	-65
Ibuprofen	0.995	2-70	5.16	1.70	85 ± 5	-30
Naproxen	0.998	0.2-25	2.04	0.67	70 ± 16	-40
Trimethoprim	0.995	0.2-5	0.16	0.05	36 ± 4	+208

### Pharmaceuticals in Untreated Urine

As pharmaceutical removal was leading in determining viable treatment conditions, pharmaceutical concentrations were first determined in untreated urine. Of the target pharmaceutical analytes, only ibuprofen, naproxen, and hydrochlorothiazide (HCT) were detected. Ibuprofen (465 ± 49.8 ng mL<sup>-1</sup>) and naproxen (172 ± 48.4 ng mL<sup>-1</sup>) showed high concentrations. Both are common non-steroidal anti-inflammatory drugs (NSAIDs), available for purchase without prescription at any drug store in the Netherlands. HCT – a commonly prescribed diuretic – was only detected in minor amounts (2.18 ± 0.24 ng mL<sup>-1</sup>). Diuretics are used to a much lower degree than NSAIDs, explaining the lower concentration as compared to the detected NSAIDs. Furthermore, HCT is known to degrade over time in urine under anaerobic conditions at pH 9 via hydrolysis.<sup>55</sup> While these conditions differ from the ones employed in this work, degradation during storage remains a possibility.

### Pharmaceutical Removal by PAC Treatment

A recent study by Duygan *et al.* (2021) showed the efficacy of using PAC for pharmaceutical removal in spiked source-separated urine, reporting over 90% removal of 12 pharmaceuticals after 24 h using 200 mg L<sup>-1</sup> PAC.<sup>55</sup> Concentrations of those pharmaceuticals were similar to those found in our samples, but they were different compounds. In a different study by Mailler *et al.* (2015), it is stated that ibuprofen adsorbs relatively poorly to PAC.<sup>93</sup> As this study aims for complete pharmaceutical removal – notably ibuprofen – using short treatment times, investigation of PAC treatment commenced using a loading of 2 g L<sup>-1</sup>. Under these conditions, all three detected pharmaceuticals present in the untreated urine were almost completely removed after 1 h of mixing. Hydrochlorothiazide was not detected and naproxen found to be below LOD. Naproxen was not detected anymore after 6 h of mixing. However, ibuprofen was consistently detected even after 24 h, albeit below LOQ at all time intervals (Table 5). Increasing the loading to 3 g L<sup>-1</sup> showed detected analytes to be completely removed in 1 h (Table 5). The short contact time required can be attributed to PAC's high surface area and high dispersion by mixing. Yet, its physical appearance as a fine powder makes it a material difficult to handle, and post-treatment filtrations proved cumbersome. Attempts at using PAC in a column failed due to immediate frit clogging and consequent negligible flowrate, making PAC not ideal for larger scale applications.

Table 5. Results for PAC treatment experiments. nd = not detected.

Analyte	Initial concentration (ng mL <sup>-1</sup> )	2 g PAC L <sup>-1</sup> (ng mL <sup>-1</sup> )		3 g PAC L <sup>-1</sup> (ng mL <sup>-1</sup> )	
		1 h	6 h	24 h	1 h
Ibuprofen	465 ± 48.4	< LOQ	< LOQ	< LOQ	nd
Naproxen	172 ± 49.8	nd	nd	nd	nd
Hydrochlorothiazide	2.18 ± 0.24	nd	nd	nd	nd

### Pharmaceutical Removal by GAC Treatment

Next, we compared PAC with GAC and used initial column loadings for GAC treatment of 2 g L<sup>-1</sup>. Two different percolation rates were investigated to assess the influence of residence time, corresponding to 6 and 18 h running time. Both ibuprofen and naproxen were still present in high concentrations of 304 ± 34.5 and 107 ± 31.6 ng mL<sup>-1</sup>, respectively (Figure 1). Note that hydrochlorothiazide is not shown in Figure 1 as it was not detected in any of the samples. Interestingly, lower percolation rate appeared to have little effect on naproxen removal, whereas ibuprofen adsorption was significantly higher. Hydrophobicity – represented by log K<sub>ow</sub> – is an important factor in assessing the adsorption tendency of micropollutants, influencing the adsorption capacity of micropollutants onto GAC.<sup>48,56</sup> Hence, a possible explanation for the different response between naproxen and ibuprofen could be the difference in log K<sub>ow</sub>. Ibuprofen has a log K<sub>ow</sub> of 4.13 whereas naproxen sits at 3.24, making ibuprofen the more lipophilic of the two. While others have found no correlation between log K<sub>ow</sub> and pharmaceutical adsorption rates, those conclusions were drawn using PAC and varying AC loading instead of contact time.<sup>55,70</sup> This result also points towards an interplay between advection and diffusion within the system, where diffusion can be regarded as analyte penetration into the porous structure of GAC. As aqueous diffusion coefficients of ibuprofen are in the order of 1·10<sup>-10</sup> m<sup>2</sup> s<sup>-1</sup> – many orders of magnitude smaller than accompanying advection rates in such systems – lower percolation rates will virtually always allow for more diffusion and thus adsorption, not taking into account possible saturation of the adsorbent surface.<sup>94</sup> This appears to hold especially true for more hydrophobic species, although a broader dataset would be warranted to state this with more certainty.

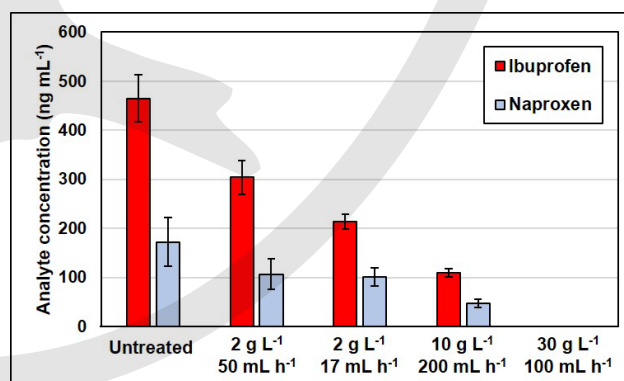


Figure 1. Ibuprofen and naproxen concentrations after GAC treatment, using different loadings and percolation rates.

An adaptation to the setup, using a higher loading of  $10 \text{ g L}^{-1}$  GAC and  $200 \text{ mL h}^{-1}$  as percolation rate showed significantly improved removal at about 75% for both analytes (Figure 1). This indicates that increased loading and column length are preferred over longer residence time for pharmaceutical removal – although both play an important role. Finally, complete removal of both analytes was achieved using  $30 \text{ g L}^{-1}$  at a percolation rate of  $100 \text{ mL h}^{-1}$ . To be able to truly make a predictive curve on pharmaceutical removal as a function of AC loading and percolation rate, detailed information on diffusion rate, advection rate and adsorption tendency per analyte is needed. As these are also dependent on the matrix of the system, this would require significant resources to elucidate and was therefore deemed outside the scope of the current study.

### Considerations on AC Treatment

GAC column treatment was preferred over PAC mixing due to ease of handling, implementation and scale-up as well as the potential for continuous treatment. Important to consider is the regeneration of AC. Many studies have investigated this, and it is possible to extend AC lifetime by steam, microwave, thermal and chemical means, among others.<sup>51,95–97</sup> In some cases, adsorptive capacities were shown to be increased after regeneration.<sup>96</sup> Should the used AC become truly depleted however, incineration will likely prove necessary as is the case for AC used in hazardous contexts.<sup>51</sup> An interesting alternative novel adsorbent is biochar, a porous carbonaceous material produced by thermochemical treatment of biomass in an oxygen-deprived environment (pyrolysis).<sup>98</sup> It is considered to be a more sustainable material than AC, especially when produced from lightly contaminated waste such as sewage sludge and should thus be considered when investigating adsorption of contaminants.<sup>99</sup> However, it does show significantly lower surface areas which is why AC was chosen over biochar in this particular study.

### Suspect Screening

To thoroughly assess the efficacy of AC treatment, urine samples of both before and after complete pharmaceutical removal were subjected to suspect screening. For untreated urine, this yielded 95 unique hits with confidence level 4 and above (see SI Figure S2 on confidence levels). This was narrowed down to 13 hits by manual investigation of MS/MS data for confidence levels 3 and above (Figure 2), while hits with confidence level 4 were not taken into further consideration. Of the three pharmaceuticals detected using target screening, initially only naproxen was tentatively identified during suspect screening. An explanation for the absence of ibuprofen was found after inspecting the extracted chromatogram ( $m/z = 205.1234 \pm 0.005$ ) using *DataAnalysis* software. In the data-dependent acquisition method required for subsequent suspect screening, ibuprofen's qualifier ion ( $m/z 161.1330$ ) cannot be detected. Lacking the MS/MS data required for a high enough confidence level, the feature was removed during the workflow. Ibuprofen's presence is certain as it was found during target analysis, therefore ibuprofen was incorporated in figure 2. Its metabolite 1/2-hydroxyibuprofen did show up in the suspect screening, corresponding to a significant feature intensity second only to caffeine and ibuprofen itself.

HCT was only found in low concentrations during target screening, so the possibility of it having been filtered out in the suspect screening workflow was checked manually. Indeed, extraction of the relevant chromatogram ( $m/z = 295.9572 \pm 0.005$ ) showed the intensity of the corresponding peak to be 3024, whereas an intensity threshold of 10000 was used. For the sake of consistency with regard to other compounds which may also have been excluded due to this threshold, HCT was not included in figure 2.

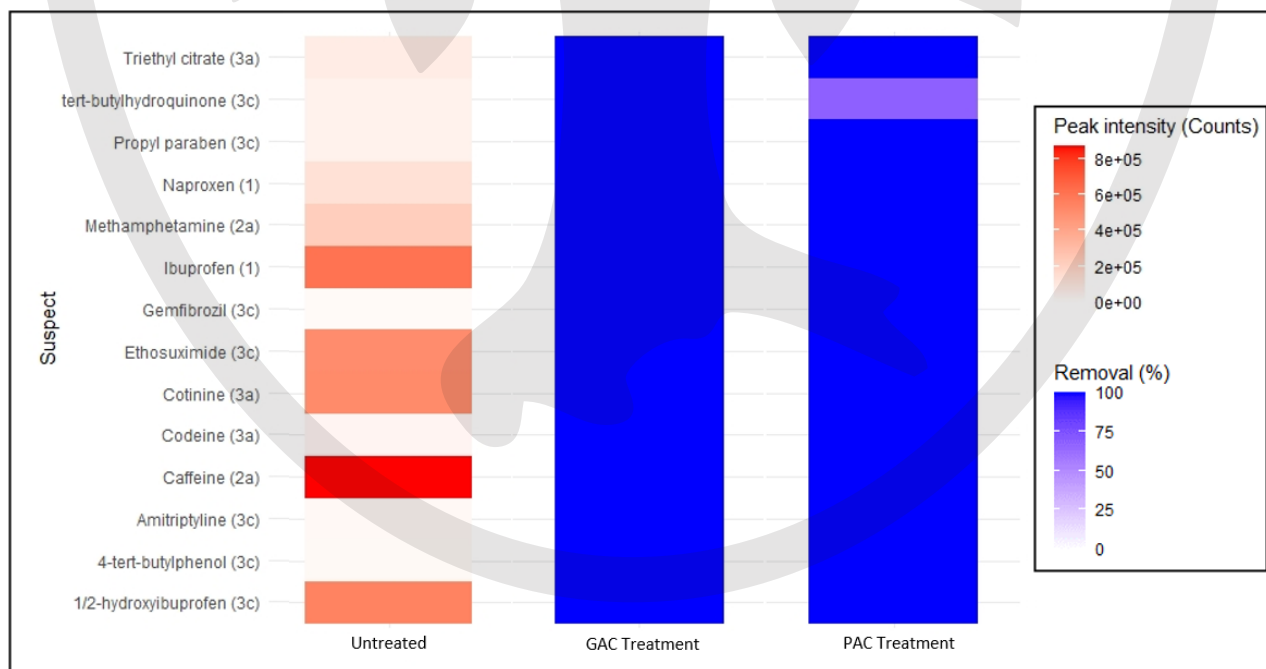


Figure 2. Tentatively identified compounds in untreated, GAC- and PAC treated urine, with identification levels in brackets.



The other tentatively identified suspects can be divided into food additives, pharmaceuticals and – more roughly – stimulants. In the case of food additives, triethyl citrate (E1505), *tert*-butylhydroquinone (E319) and propyl paraben (E216) were found. Identified pharmaceuticals – other than the ones discussed above – were codeine (opioid), ethosuximide (anticonvulsant), amitriptyline (antidepressant) and gemfibrozil (a cholesterol-lowering drug). Grouped as “stimulants”, caffeine, cotinine (nicotine metabolite) and methamphetamine (drug of abuse) were tentatively identified. Perhaps unsurprising, caffeine showed the highest intensity count out of all suspects.

The only tentatively identified suspect which could not be readily placed in one of these categories is 4-*tert*-butylphenol. Most commonly used in the curing of epoxy resins, as plasticizer, and as surfactant, its presence in urine is less easily explained as compared to the other suspects.<sup>100</sup> Though, its presence in urine has been confirmed in a previous study, showing the plausibility of the suspect.<sup>101</sup> Figure 2 lists all tentatively identified suspects and their respective peak intensity counts, as well as their removal rates by AC treatment. Identification levels for each suspect are given in brackets. After PAC and GAC treatment, suspect screening yielded 7 and 2 hits, respectively, brought back to only *tert*-butylhydroquinone being tentatively identified after manual inspection. As an isolated case, this sole suspect was not completely removed after PAC treatment, with a removal rate of 69%.

Three other chemicals were identified but not incorporated into the final list of tentatively identified suspects. Firstly, diethyl phthalate was found in untreated urine as well as both PAC- and GAC-treated urine. However, it is a commonly-used plasticizer and results indicated higher concentrations after AC treatment than before. Hence, it was concluded that the suspect was likely leached from plastics used in sample preparation and handling. Second- and thirdly, (S)-mandelic acid and 4-hydroxyphenylacetic acid were tentatively identified but not part of the NORMAN suspect list. This is due to nature of the suspect screening workflow: in these cases, the compounds were part of the candidates obtained with MS/MS compound annotations, for which at least one candidate was part of the suspect list. Upon inspection, the MS/MS data pointed towards candidates 2<sup>nd</sup> and 5<sup>th</sup> in rank for their respective features, which were not part of the suspect list and therefore not further taken into account. See SI Excel S1 and S2 for detailed information on suspects found in negative and positive mode, respectively.

These results highlight the efficacy of both AC treatments; not only were all the tentatively identified suspects removed except one in the case of PAC (*tert*-butylhydroquinone), the initial hits were reduced from 95 to 7 and 2 for PAC and GAC treatment, respectively. While these initial hits were not all tentatively identified, they do correspond with species in the urine matrix which were subsequently removed.

$$\text{Fertilizing component in dry matter (\%)} = \frac{\text{Nutrient content (\%)}}{\text{Dry matter content (\%)}} \times 100\% \quad (\text{Eq. 3})$$

### Influence of AC Treatment on Nutrient Content

While recent work has shown that nutrient content is not significantly or only slightly affected by AC treatment,<sup>49,102</sup> the relatively high loadings used in this study could adversely affect nutrient concentrations to a higher degree. As such, selected urine samples were measured in triplicate for N and P content pre- and post-treatment. GAC treatment showed a similar reduction in both N and P content at 16.3% and 16.7%, respectively, where initial concentrations were  $2426 \pm 53 \text{ mg L}^{-1} \text{ N}$  and  $817 \pm 84 \text{ mg L}^{-1} \text{ P}_2\text{O}_5$ . PAC treatment had significantly less impact on nutrient concentrations, with only 4.5% N and 1.4% P removed (Figure 3). This significant difference can be ascribed to the different AC loadings of  $30 \text{ g L}^{-1}$  GAC versus  $3 \text{ g L}^{-1}$  PAC, as well as shorter residence time in the case of the latter. Hence, while GAC offers much easier treatment in terms of handling, the removal of nutrients using this material with the chosen parameters is apparent and should be taken into account when aiming to produce fertilizer from urine at scale.

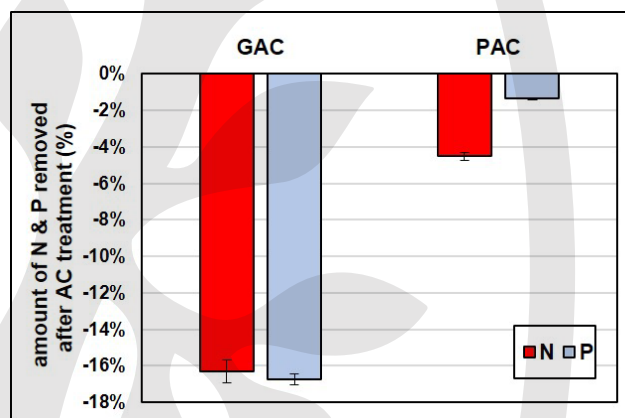


Figure 3. P & N removal by AC treatment.

### Closing the Loop: A Dutch fertilizer

The removal of organic contaminants of concern using AC treatment has now been established. Yet, for a urine-based fertilizer – or any novel fertilizer for that matter – to qualify as legal in the Netherlands, it needs to adhere to legal limits regarding fertilizing components, inorganic contaminants and POPs. Results verifying this adherence are discussed in the following paragraphs.

### Fertilizing Components

As mentioned, adherence to fertilizing components requires the dry matter of the respective liquid material to contain 0.5% wt. of either N,  $\text{P}_2\text{O}_5$  or  $\text{K}_2\text{O}$ . In this study, N and  $\text{P}_2\text{O}_5$  were focused on in particular. Dry matter content was determined according to EN 15934:2012 (see SI Table S4) and found to be  $0.575\% \pm 0.003\%$ . Based on eq. 3, the fertilizing components N and P in dry matter after AC treatment were calculated and are given in Table 6. Two datasets were used for this; values given are means and standard deviations are included. In both cases, the fertilizing component in dry matter adheres with ease to the legal minimum required for a material to be considered as fertilizer in the Netherlands:  $11.9\% \pm 1.49$  and  $35.4\% \pm 0.81$  for  $\text{P}_2\text{O}_5$  and N, respectively.

Table 6. Determination of fertilizing components in dry matter (%) based on urine dry matter content and nutrient contents.

Fertilizing component	Required fertilizing component in dry matter (%)	Fertilizing component in liquid urine (%)	Fertilizing component in dry matter (%)
P <sub>2</sub> O <sub>5</sub>	0.5	0.068 ± 0.009	11.9 ± 1.49
N	0.5	0.20 ± 0.005	35.4 ± 0.81

### Inorganic Contaminants

Inorganic contaminants (HMs) are defined as a function of the most abundant fertilizing component – in this case N. HM contents per N (mg kg<sup>-1</sup>) were determined based on the same datasets as above and offset against their legal limits in Table 7. It should be noted that the given HM concentrations (second column) represent untreated urine, as HMs were not expected to be removed during AC treatment and therefore not measured post-treatment. However, the N concentration was corrected for N removal by GAC treatment (-16.3%) as to establish a lower N limit. The HM content per N can thus be seen as an upper limit and worst-case scenario for these particular datasets. In all cases, HM content per N was far below the maximum allowed legal limit.

### POP Screening

Adhering to maximum allowable POP concentrations is the final prerequisite for a fertilizer material to be legally eligible in the Netherlands. POPs in question include OCPs, PCBs and PAHs, which were not included in the suspect screening. Due to the lipophilicity of these compounds, they tend to accumulate in body fat rather than being excreted.<sup>103</sup> As such, their presence in urine was not expected – especially following AC treatment. Screening for POPs was therefore done in a qualitative manner. Indeed, none of the screened POPs listed were detected in AC-treated urine (SI Table S5). Polychlorinated dibenzodioxins (PCDDs) and -furans (PCDFs) were not screened in this process due to budgetary constraints. They do, however, have the strictest limit with an allowable concentration of all POPs at 15 µg Σ PCDD/PCDF kg N<sup>-1</sup>, which invites further discussion. In a recent study by Han *et al.* (2022), Σ<sub>27</sub> PCDD/PCDF concentrations were determined at 1640–5415 pg L<sup>-1</sup> in blood samples from people living in industrialized areas in China prone to dioxin emissions.<sup>104</sup> Considering a hypothetical scenario where a person at the upper limit of said PCDD/PCDF concentrations would excrete it all in one urinary session, this would result in about 14–27 µg Σ PCDD/PCDF kg N<sup>-1</sup>; at or slightly above the legal limit (see SI Excel S3 for assumptions made and data used). As this scenario is far from realistic, we can assume with reasonable confidence that dioxins will not pose a problem in adhering to legal fertilizer standards. Lastly, mineral oil was also not taken into account due to its very high permissible concentration of 748 g kg N<sup>-1</sup>. Clearly, this limit was set for fertilizer materials originating from different sources and is not possible to reach in a urine-based fertilizer. As such, all prerequisites to apply for fertilizer- and EoW status are met for AC-treated urine, paving the way for its legal use as fertilizer in the Netherlands.

Table 7. HM concentrations in untreated urine, offset against N content and their legal limits.

Inorganic contaminant	Concentration (µg L <sup>-1</sup> )	HM content per N (mg HM kg N <sup>-1</sup> )	Max. HM allowed per N (mg HM kg N <sup>-1</sup> ) <sup>62</sup>
Cd	< 0.1	< 0.05	25
Cr	36.6 ± 11.1	18.00 ± 5.46	1500
Cu	35.4 ± 12.5	17.38 ± 6.12	1500
Hg	< 0.05	< 0.02	15
Ni	3.3 ± 0.15	1.60 ± 0.07	600
Pb	0.21 ± 0.04	0.10 ± 0.02	2000
Zn	15 ± 0.02	7.38 ± 0.10	6000
As	0.99 ± 0.52	0.48 ± 0.25	300

### Conclusions

In this study, we set out to provide proof-of-principle for the possible production of a biobased fertilizer from source-separated urine using AC adsorbents, with the use of national (Dutch) fertilizer legislation as guiding framework and the application of additional suspect screening as notable novelties in this field. Three of the eight target pharmaceuticals were detected pre-treatment, of which common NSAIDs ibuprofen and naproxen were found in high concentrations. Furthermore, 11 contaminants of concern were tentatively identified using the NORMAN list of priority contaminants consisting of 976 substances. After 1 h of mixing with 3 g L<sup>-1</sup> PAC, only one suspect was still detected. Treatment with 30 g L<sup>-1</sup> GAC using a column with a running time of 3 h completely removed all identified substances. Screening for inorganic contaminants and persistent organic pollutants – in line with Dutch fertilizer legislation – showed all further requirements for fertilizer status to have been met. The high AC loadings employed did however affect the nutrient concentrations, especially using the GAC treatment with about 16% of both N and P removed. Still, GAC treatment is the preferred treatment method for further scale up and implementation due to its significantly easier operation. Scaling up this method would be an interesting avenue for future investigation and should be explored to allow for the use of urine as biobased fertilizer in meaningful quantities. Subsequent investigations into the longevity and regeneration potential of the used GAC material could then shed further light on the large-scale potential of this method.

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### Primary Data

10.5281/zenodo.10992983

### Conflict of Interest

The authors declare no conflict of interest.

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## Supplementary information

### ***Urine As Biobased Fertilizer: The Netherlands as Case Study***

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##### **Accompanying excel file:**

S1. Detailed information on found suspects in negative mode

S2. Detailed information on found suspects in positive mode

S3. Back-of-the-envelope worst-case scenario for PCDD/PCDF excretion

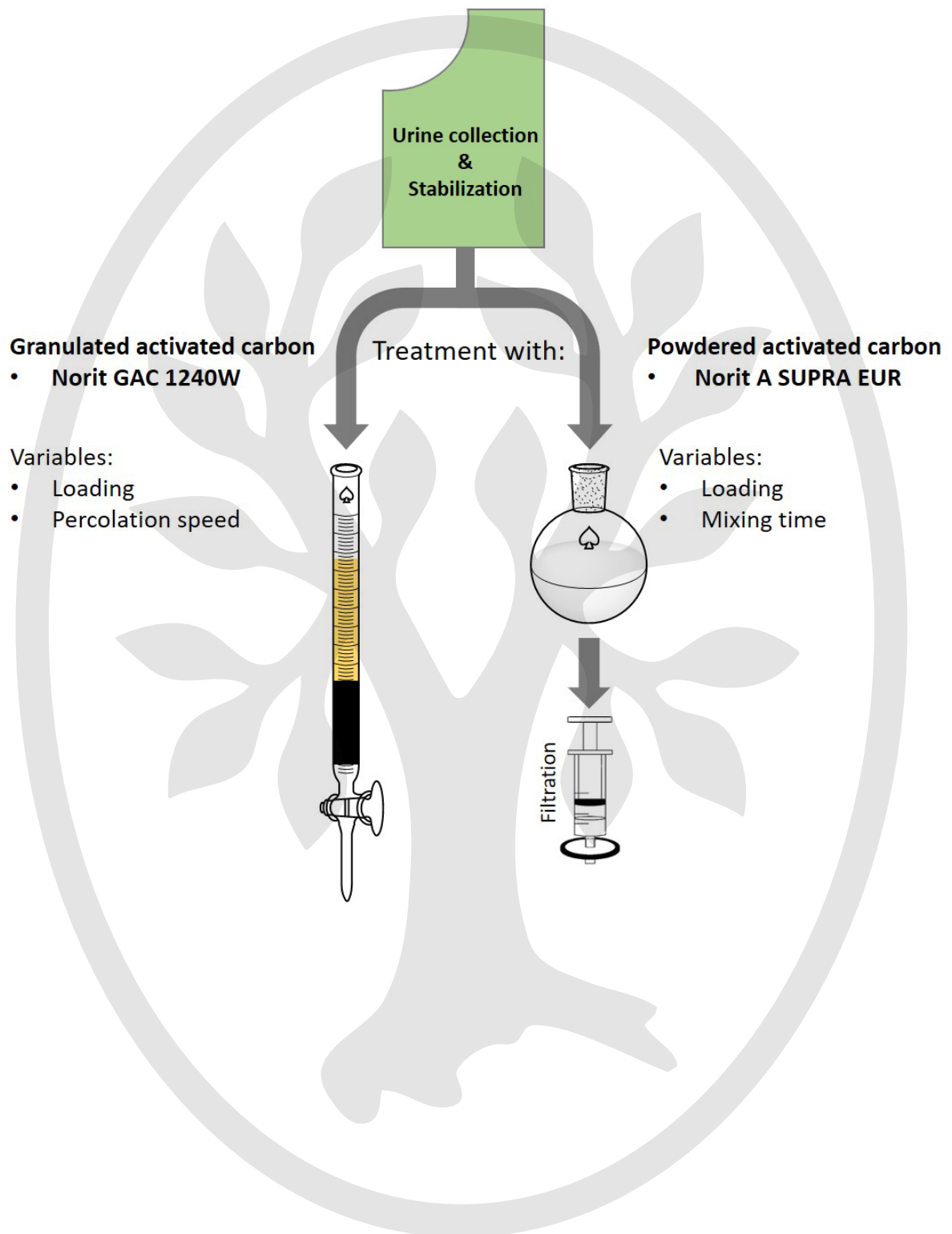
**Table S1. Detailed information on reference standards and auxiliary chemicals**

	<b>Name</b>	<b>Uses</b>	<b>Supplier</b>	<b>CAS</b>
Reference standards	Atenolol	Beta blocker	Sigma Aldrich	29122-68-7
	Hydrochlorothiazide	Diuretic	Sigma Aldrich	58-93-5
	Diclofenac	NSAID	Sigma Aldrich	15307-86-5
	Sulfamethoxazole	Antibiotic	Sigma Aldrich	723-46-6
	N4-Acetylsulfamethoxazole	Metabolite of SMX	Sigma Aldrich	131549-85-4
	Ibuprofen	NSAID	Sigma Aldrich	15687-27-1
	Naproxen	NSAID	Sigma Aldrich	22204-53-1
	Trimethoprim	Antibiotic	Sigma Aldrich	738-70-5
Auxiliary chemicals	Methanol (>99.9%)	Solvent	Biosolve	67-56-1
	Formic acid (99%)	Sample acidification	Biosolve	64-18-6
	Sodium acetate	Mobile phase modifier	Merck	127-09-3

**Table S2 Activated carbon specifics**

	<b>Norit A SUPRA</b>	<b>Norit GAC 1240 W</b>
<b>Type</b>	Powdered	Granulated
<b>Surface area (m<sup>2</sup> g<sup>-1</sup>)</b>	1700	1100
<b>Iodine number</b>	1550	min. 950
<b>pH</b>	Neutral	Alkaline
<b>Particle size</b>	D <sub>10</sub> : 4 μm	D <sub>5</sub> : 0,425 mm
	D <sub>50</sub> : 20 μm	D <sub>90</sub> : 1,7 mm
	D <sub>90</sub> : 100 μm	

Figure S1 Cleaning process diagram



**Table S3 list of quantifier and qualifier ions for selected analytes**

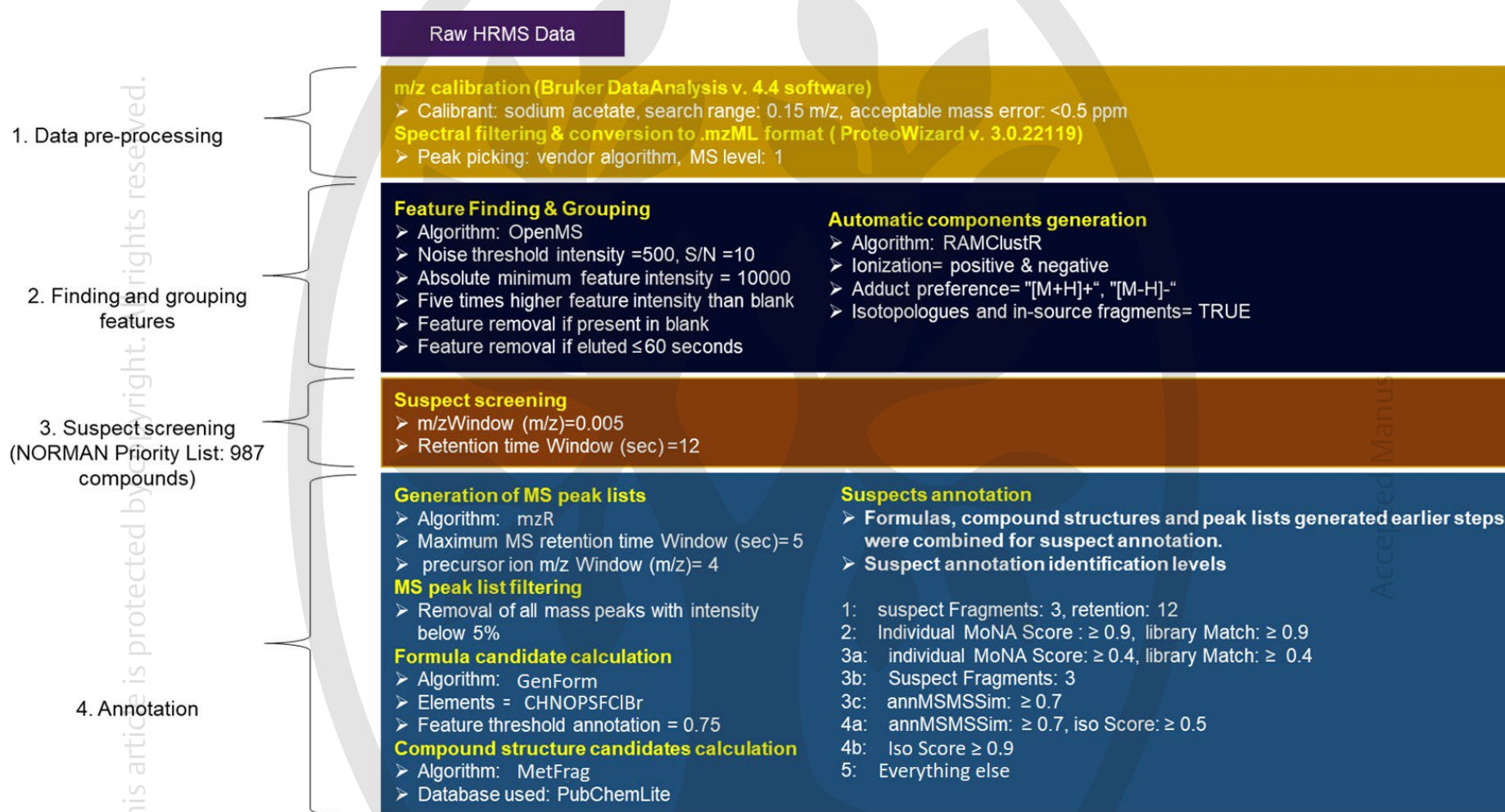
Compound name	Formula	Quantifier m/z (Q)	Qualifier m/z (q)	q/Q	ESI mode	RT	Internal standard
Atenolol	C <sub>14</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub>	267.1703	268.1747	1.00	+	8	Atenolol-d7
Hydrochlorothiazide	C <sub>7</sub> H <sub>8</sub> ClN <sub>3</sub> O <sub>4</sub> S <sub>2</sub>	295.9572	268.9492	0.91	-	8.30	n/a
Diclofenac	C <sub>14</sub> H <sub>11</sub> Cl <sub>2</sub> NO <sub>2</sub>	294.0094	250.0187	0.85	-	15.98	Diclofenac-13C6
Sulfamethoxazole	C <sub>10</sub> H <sub>11</sub> N <sub>3</sub> O <sub>3</sub> S	254.0594	162.0316	0.64	+	11.49	Sulfamethoxazole-13C6
N4-Acetylsulfamethoxazole	C <sub>12</sub> H <sub>13</sub> N <sub>3</sub> O <sub>4</sub> S	294.0554	198.0226	0.67	-	11	Sulfamethoxazole-13C6
Ibuprofen	C <sub>13</sub> H <sub>18</sub> O <sub>2</sub>	205.1234	161.1330	0.79	-	14.8	Ibuprofen-d3
Naproxen	C <sub>14</sub> H <sub>14</sub> O <sub>3</sub>	231.1016	170.0711	0.74	+	15.23	Naproxen-methoxy-d3
Trimethoprim	C <sub>14</sub> H <sub>18</sub> N <sub>4</sub> O <sub>3</sub>	291.1452	313.1271	1.08	+	10	Trimethoprim-d9

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Figure S2 Detailed parameters suspect screening (taken and adapted from Das *et al.*, 2023).<sup>1</sup>



### Text S1. Dry matter content determination of urine samples

100 mL of the urine matrix was added to a 250 mL round-bottom flask and weighed. The flask was then attached to a BUCHI Rotavapor® R-100. Pressure was set to 60 mbar and bath temperature to 40 °C. After most of the water was evaporated, the flask was put into a heating bath at 105 °C and left to dry overnight without a stopper.<sup>2</sup> The flask was subsequently weighed, and the resulting weight subtracted from the initial weight. This number was then offset to the density of the urine, which was assumed to be 1.010 g / mL. Obtained numbers are given in Table S 4.

Table S4. Dry matter content of urine samples

Replicate	Residual weight (mg)	Dry matter content (%)
1	581.4	0.576
2	578.2	0.572
3	583.5	0.578
		0.575 ± 0.002

**Table S5. Results for POP screening of AC-treated urine offset against legal limits.**

POP	Legal limit (mg kg N <sup>-1</sup> ) <sup>3</sup>	PAC-treated urine	GAC-treated urine
Σ PCDD/PCDF	0.015	Not investigated	Not investigated
α-HCH	248	nd	nd
β-HCH	9.6	nd	nd
γ-HCH (lindane)	0.96	nd	nd
HCB	31.2	nd	nd
Aldrin	5.6	nd	nd
Dieldrin	5.6	nd	nd
Σ Aldrin/Dieldrin	5.6	nd	nd
Endrin	5.6	nd	nd
Isodrin	5.6	nd	nd
Σ Endrin/Isodrin	5.6	nd	nd
Σ DDT + DDD + DDE	18.6	nd	nd
PCB-28	14.8	nd	nd
PCB-52	14.8	nd	nd
PCB-101	60	nd	nd
PCB-118	60	nd	nd
PCB-138	60	nd	nd
PCB-153	60	nd	nd
PCB-180	60	nd	nd
Σ 6-PCB (excl. PCB-118)	300	nd	nd
Naphtalene	480	nd	nd
Fenanthrene	600	nd	nd
Anthracene	480	nd	nd
Fluoranthene	148	nd	nd
Benzo(a)anthracene	184	nd	nd
Chrysene	184	nd	nd
Benzo(k)fluoranthene	216	nd	nd
Benzo(a)pyrene	232	nd	nd
Benzo(g,h,i)perylene	168	nd	nd
Indeno(1,2,3-c,d)pyrene	188	nd	nd
Σ 10-PAK	9200	nd	nd
Mineral oil	748000	Not investigated	Not investigated

nd = not detected.

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