Removal and recovery of N, P and K from urine via ammonia stripping and precipitations of struvite and struvite-K
Kangning Xu, Chi Zhang, Jiyun Li, Xiang Cheng and Chengwen Wang

ABSTRACT

This study investigated the recovery of N, P and K from source-separated urine in laboratory-scale combined processes of air stripping and precipitation. Two operation scenarios were carried out to recover N/P (named partial scenario) and N/P/K (named complete scenario). Most of the nutrients were recovered (> 70%) by optimising the operation parameters including the increase of air flow rate and more dosages of the sources of Mg and P. Absorbent rich in ammonium sulphate and solid precipitates including struvite, struvite-K, and struvite-Na was produced. The simultaneous recovery of P and K was the key process to determine the substance input. The ratio of substance input to nutrient recovered (P2O5 and K2O) was 4.14 in the partial scenario, whereas it increased to 10.61 in the complete scenario. The inevitable co-precipitation of struvite-Na mainly responded for the lower economic efficiency of the complete scenario.

Key words | magnesium potassium phosphate hexahydrate, nutrient recovery, source-separated urine ammonia stripping, struvite precipitation

INTRODUCTION

Sewage source-separation is considered to be a sustainable option for a future sewerage system because it presents several advantages to manage resources including water, nutrients, and organics (Lienert & Larsen 2010). Human urine as the main contributor of N and P to municipal wastewater contains large amounts of nutrients. Recycling or recovery of these nutrients from source-separated urine to close the nutrient loop is one of the most attractive benefits. Particularly, this will provide new sources of phosphate fertiliser as a non-substitutable nutrient for agriculture, which will probably last for only approximately 50–100 years because of the limited exploitable phosphate reserves and increasing demand to support the global population expansion (Gilbert 2009; Hao et al. 2011).

Many processes for urine treatment have been developed, most of which mainly focused on the recovery of N and P (Maurer et al. 2006). K is also an important fertiliser for plant although it is not an aquatic pollutant. Potash ore as the main raw material for K fertiliser distributes extremely unevenly around the world (US Geological Survey 2014). Countries lacking in potash ore, including China, the USA, India, and Brazil, supplied more than half of the global food demand in 2010 (Food and Agriculture Organization 2011). Therefore, this phenomenon might induce agriculture risk for these countries as well as for the efforts addressing the global hunger. Urine accounts for approximately 80%–90% of the total K in the municipal wastewater (Larsen & Gujer 1996). This condition means that recovering K from urine would be more feasible compared with that from the greatly diluted municipal wastewater. Our recent studies showed that the precipitation of magnesium potassium phosphate hexahydrate (struvite-K, MgKPO4·6H2O) is an effective method to recover simultaneously K and P from urine (Xu et al. 2011). The precipitates obtained are also valuable slow-release fertiliser similar to magnesium ammonium phosphate hexahydrate (struvite, MgNH4PO4·6H2O). Thus, all the three macronutrients in urine might be recovered through combination of the struvite-K precipitation and an ammonium recovery process, providing a perspective for the complete recovery of N, P and K from source-separated urine.

The objective of this study is to optimise the operation of combined processes of ammonia stripping-absorption and precipitations of struvite/struvite-K to recover most of the
nutrients (>70%) from source-separated urine in laboratory scale continuous-operating reactors. The reactor performances, including the efficiency of nutrient removal/recovery and the nutrient products, were evaluated in two operating scenarios to recover N/P and N/P/K, respectively. The results would provide a technical option to further develop urine treatment processes.

**MATERIAL AND METHODS**

**Urine preparation**

Pure urine was collected from several volunteers and then stored in polyvinyl chloride tanks (20 L). Stale urine was inoculated at a volume ratio of approximately 15% to accelerate the ammonification of pure urine according to a reported method (Liu et al. 2008a). The urine with full ammonification before being used in the experiments was diluted thrice in an influent tank (60 L) with deionised water to simulate the actual diluted urine with flushing water. New urine influent was supplied when the diluted urine was used up in the influent tank. Characteristics of the influent urine analysed per batch are summarised in Table 1.

**Experimental procedures**

Combined processes for the nutrient recovery were established, including a stripping column, an absorption column, and a precipitation reactor (Figure 1). The size of the stripping column is similar to the absorption column at a total volume of 15 L. A heater (Anubias A9100, China) was immersed in the urine solution of the stripping column to maintain a temperature of 35 °C. The precipitation reactor exhibited a reaction and settlement zones at effective volumes of 0.5 and 2 L, respectively, similar to that reported in our previous studies (Xu et al. 2012). Two pH probes (Hach Sension-1, USA) were placed in the stripping column and the reaction zone of the precipitation reactor, respectively, to control the pH values on set points.

Two experimental scenarios were conducted in laboratory scale. A partial scenario (operation I as listed in Table 2) was carried out mainly to remove and recover N and P from urine. Herein, the urine influent was firstly pumped into a precipitation reactor where a solution source of Mg (2 M MgCl₂·6H₂O) was added. The outflow was then pumped into the stripping column for further treatment. The complete recovery of N, P and K was further investigated (named as complete scenario). In this scenario, the urine influent was firstly stripped in the stripping column, followed by the treatment in the precipitation reactor where both sources of Mg (2 M MgCl₂·6H₂O) and P (1 M Na₂HPO₄·12H₂O) were added. The effect of air flow was compared in operations II and III, whereas the effect of Mg:K:P molar ratio was studied in operations III and IV (details in Table 2).

The stripping column and the precipitation reactor were operated continuously at an influent flow rate of 0.25 L h⁻¹. Solution volume in the stripping column was maintained at approximately 6 L. Sulphuric acid (2 M) was employed as absorbent for ammonia gas in the air spilled out from the stripping column using an air compressor (Lida Z-0.12/8, China). The absorbent enriched with ammonium was emptied. Fresh absorbent (8 L) was added in the absorption column when the absorption reached equilibrium. Precipitate slurry formed was discharged from the bottom of the precipitation reactor at a crystal retention time of 10–12 h (defined in Xu et al. 2012). After which, the slurry was dewatered and the precipitates were collected for further analysis. All solution samples were taken at a volume of 5 mL. Then, a drop of 1 M HCl was added to the samples prior to analysis.

**Analytical methods**

Concentrations of chemical oxygen demand (COD), ammonium, and phosphate in solution samples were analysed colourimetrically on a spectrophotometer (Lianhua Technology 5B-3B (V8), China) according to standard methods (Ministry of Environmental Protection 2006). Contents of cation including K, Na, Ca and Mg were measured...
by inductively coupled plasma-atomic emission spectroscopy (Thermo, USA). Removal efficiencies of N, K and P were calculated on the basis of element concentrations in the influent and the effluent of the reactor. Recovery efficiency of nutrient was estimated by the ratio of the amount in the nutrient products obtained to the total amount in the influent.

Up to 50 mL slurry collected was filtrated using 0.45 μm filters and then dried in an oven at 50 °C for 48 h before the mass weighing to calculate the precipitate concentration. Crystalline form of the dried precipitates was assayed using X-ray diffractometer (XRD, Rigaku TTR-III, Japan) using a Cu anode at a wavelength of 0.14506 nm and a

![Figure 1](https://iwaponline.com/wst/article-pdf/75/1/155/456610/wst075010155.pdf)

**Table 2**  Operating parameters of the combined processes

<table>
<thead>
<tr>
<th>Reactors</th>
<th>Parameters</th>
<th>Operation I</th>
<th>Operation II</th>
<th>Operation III</th>
<th>Operation IV</th>
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<tr>
<td>Stripping column</td>
<td>pH</td>
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<td>10.6 – 11</td>
<td>10.6 – 11</td>
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<td></td>
<td>Air flow (m³ h⁻¹)</td>
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<td>1.0</td>
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<td>1.5</td>
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<td></td>
<td>Influuent (L h⁻¹)</td>
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<td></td>
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<tr>
<td>Absorption column</td>
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<td>8</td>
<td>8</td>
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<tr>
<td></td>
<td>H₂SO₄ (M)</td>
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<td>2</td>
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<tr>
<td>Precipitation reactor</td>
<td>pH</td>
<td>No control</td>
<td>10.4 – 10.6</td>
<td>10.4 – 10.6</td>
<td>10.4 – 10.6</td>
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<tr>
<td></td>
<td>Stirring (rpm)</td>
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<td>300</td>
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<tr>
<td></td>
<td>Mg:K:P ratio</td>
<td>1.1:1⁺</td>
<td>3:1:3</td>
<td>3:1:3</td>
<td>3.5:1:3</td>
</tr>
</tbody>
</table>

⁺Mg:P ratio.

![Absorption column](https://iwaponline.com/wst/article-pdf/75/1/155/456610/wst075010155.pdf)
scanning rate of 6 min⁻¹. Morphology and elements of the dried precipitates were determined on a scanning electron microscopy/energy dispersive spectrometer (SEM-EDS, Cambridge S-360, UK) at an emission voltage of 20 kV.

RESULTS AND DISCUSSION

Nutrient removal in the partial scenario

The ammonium in the diluted urine was firstly removed via the precipitation of struvite and then stripped out in the stripping column in the partial scenario. The concentration of ammonium on average decreased to 153 ± 41 mg N L⁻¹ after the stripping column (Figure 2(a)). As such, this resulted in an ammonium removal efficiency of 95%. Calculations showed that the maximum amount of ammonium removed in the precipitation reactor can reach 70 mg N L⁻¹ at an influent phosphate of 99–138 mg P L⁻¹ according to the stoichiometric composition of struvite. Thus, the removal of ammonium via the struvite precipitation is lower than 3.2%.

Phosphate was removed in the precipitation reactor. The phosphate concentration in the effluent from the stripping column was 20 ± 6 mg P L⁻¹, corresponding to a total removal efficiency of 83% (Figure 2(b)). Measurements showed no significant changes in the concentrations of K before and after the precipitation reactor (absolute value of relative error <5%). Thus, this finding indicates no precipitation of struvite-K. As a result, most of N and P in the diluted urine were removed, whereas nearly no K was precipitated when the diluted urine was treated as defined in the partial scenario.

Nutrient removal in the complete scenario

The complete scenario including three operation modes was designed to recover all the three macronutrient elements in the diluted urine. After being first treated in the stripping column, the ammonium decreased on average to 169 ± 26 mg N L⁻¹ at an air flow rate of 1 m³ h⁻¹. A removal efficiency of 91% was achieved (Figure 3(a)). This result equalled to that in the partial scenario if the removal of ammonium was excluded because of the struvite precipitation. Thus, the pre-treatment in the precipitation reactor exhibited little effect on eliminating ammonium in the stripping column. The stripped urine was then pumped into the precipitation reactor and the ammonium in the effluent further decreased to 32 ± 9 mg N L⁻¹ (Figure 3(b)). This result means that approximately 80% of the ammonium in the stripped urine was removed via the precipitation of struvite. As a consequence, the total efficiency of ammonium removal reached 98% in operation II.

The stripping capacity was significantly promoted when the air flow rate was increased to 1.5 m³ h⁻¹ in the next two operation modes (III and IV). The effluent ammonium from the stripping column decreased to 26 ± 18 mg N L⁻¹, generating an average removal efficiency of approximately 99%. The residual ammonium was reduced to only 2.2 ± 1.2 mg N L⁻¹ following the treatment in the precipitation reactor. Therefore, nearly all the ammonium in the diluted urine (99.9%) was removed via the combined processes of stripping and precipitation.

Sources of Mg and P were added in the precipitation reactor at a pre-set molar ratio of Mg:K:P to maximise the precipitation of struvite-K in the stripped urine. Then, the concentration of phosphate was reduced to an average value of 62 ± 18 mg P L⁻¹ when the precipitation reactor was operated at a Mg:K:P ratio of 3:1:3 (Figure 4(a)). Herein, the removal efficiency of phosphate was only approximately 45% in operations II and III (Figure 4(b)). Dosage of the Mg source was increased in operation IV to enhance the phosphate removal. Accordingly, the effluent phosphate concentration decreased to 25 ± 9 mg P L⁻¹ when the Mg:K:P ratio was set as 3.5:1:3. As a result, approximately 79% of the phosphate in the diluted urine was removed in the combined processes.

Figure 2 | Process performance for the removal of ammonium (a) and phosphate (b) from urine in operation I. Influent concentration in the influent tank (●), effluent concentration from the stripping column (○), and total removal efficiency (×).
Compared with the influent K, the effluent K from the precipitation reactor decreased to 245 ± 87 mg L\(^{-1}\) even though the Mg:K:P ratio reached 3:1:3 when the stripping column was operated at an air flow rate of 1 m\(^3\) h\(^{-1}\) (operation II, Figure 4(c)). As such, the removal efficiency of K was only 48% (Figure 4(d)). The air flow rate was increased to 1.5 m\(^3\) h\(^{-1}\) in operation III to enhance the stripping of ammonium. As a result, the removal efficiency of K also increased to 78%. This removal efficiency was further slightly promoted (80%) when more Mg sources were dosed to increase the phosphate removal in operation IV.

**Nutrient recovery and products**

The recovery of ammonium was achieved via the absorption of ammonia gas from the stripping air into the absorbent. Both solution pH and the content of ammonium in the absorbent increased significantly after the absorption was initiated in one operating cycle of the absorption column (Figure 5). The absorption reached equilibrium after approximately 50 days. The maximum concentration of ammonium in the absorbent was approximately 26.2 g N L\(^{-1}\) at a pH of approximately 9.5. In the duration of absorption, the volume of absorbent increased from 8 L to approximately 18.5 L. This result should mainly be induced by the transportation of water from the stripping column to the absorption column. Estimations showed that the stripped amount of ammonium-N was approximately 617 g, whereas the absorbed amount reached 485 g in the first cycle of the absorption column. Thus, the absorption efficiency was estimated to be 79%. An ammonium recovery efficiency of 75% was thereby achieved.

Measurement of absorbent pH was used to assess the absorption process in next operating cycles of the absorption column. This method was considered to reach absorption equilibrium when the pH value becomes stable at approximately 9.5 for three days. Herein, the operating cycle was broken off and a new cycle was initiated with fresh absorbent. The amount of absorbent rich in ammonium sulphate was estimated to be 64 L per tons of diluted urine in this case based on operation records through the experiments. As such, ammonium in the urine was 11 times concentrated in the absorbent. The concentration factor would be further enhanced if increased concentration of H\(_2\)SO\(_4\) was employed as absorbent. This effect would reduce the transport cost and the inconvenience for fertilising operation when the absorbent rich in ammonium rather than urine was employed as liquid fertiliser.

Settlement ability of precipitates formed in the precipitation reactor is quite important for the recovery of
phosphate and K. Struvite can settle relatively fast within 1 h (Antonini et al. 2011), whereas struvite-K probably presented a different settlement behaviour (Wilsenach et al. 2007). During the four operation modes, the clearly visible interface between the solution and the slurry was nearly stable maintained at approximately 40–100 mm lower than the overflow surface. Only a very small amount of tiny precipitates flew away with the effluent. All the phenomena indicate good settlement of precipitates whether struvite or struvite-K at the given operation parameters as fully discussed and optimised in our previous studies (Xu et al. 2012).

Precipitate concentration of the slurry collected was estimated to be in the ranges from 17 g L$^{-1}$ to 28 g L$^{-1}$ after dewatering and drying. The precipitates obtained in operation I showed needle-like shape, main elements of

![Figure 4](https://iwaponline.com/wst/article-pdf/75/1/155/456610/wst075010155.pdf)
which were Mg, N, P and O with only a few amount of K (0.6%) (Figure 6(a) and 6(b)). Their XRD pattern gave typical peaks of struvite (Figure 6(c)). Thus, the dominating compound should be struvite. The production of precipitates per day reached approximately 4.1 ± 1.2 g in the partial scenario. Herein, the recovery efficiency of phosphate was estimated as approximately 81%. The precipitates collected from operation IV were much shinier and longer as shown in Figure 6(d) compared with that at other operations. EDS analysis presented the semi-quantified atomic ratio of elements Mg, K, Na, P, O and Cl to be 16.09%, 5.04%, 9.62%, 13.87%, 53.12% and 2.26%, respectively (Figure 6(e)). Then, these precipitates were confirmed to be mainly composed of struvite-K and magnesium sodium phosphate heptahydrate (MgNaPO₄·7H₂O, struvite-Na) through the XRD analysis results (Figure 6(f)). More precipitates (36 ± 7 g) were obtained per day in operation IV of the complete scenario. Estimated on a mass balance, the recovery efficiency of phosphate and K reached 72% and 73%, respectively.

**Ammonium stripping and absorption**

Stripping and absorption are commonly used processes for ammonium recovery. The process performance has also been investigated for recovering ammonium from urine in laboratory scale (Behrendt et al. 2002; Basakcilardan-Kabakci et al. 2007), pilot scale (Antonini et al. 2011) and full scale (Morales et al. 2015). Various factors including air flow rate, solution temperature, solution pH, and packing can influence the stripping efficiency. This study achieved an ammonium removal efficiency of 99% at an air flow rate of 1.5 m³ h⁻¹ when the temperature was 35 °C and the pH value of the urine solution was 10.6–11. The removal efficiency is much higher than that of many other reported values (Behrendt et al. 2002; Basakcilardan-Kabakci et al. 2007).
stripped ammonia (Antonini et al. 2011). However, air stripping generally caused high energy consumption (Antonini et al. 2011). Increased solution temperature or increased pH value should be offered when the air flow rate was reduced. These operation parameters can be further optimised by considering both the removal efficiency and the operation cost including consumption of energy and agents.

The absorption following the stripping was a key process to determine the ammonium recovery. The efficiency of ammonia absorption in the acidic solution only reached 79%. This amount represents that 21% of the ammonia in the stripping gas was lost. Many approaches might be employed to enhance the absorption of ammonia. Recirculating the gas from the absorption column filled with plastic packing (Raschig rings) back to the stripping column can lead to nearly complete absorption of the stripped ammonia (Antonini et al. 2011). Employing ceramic Raschig rings in a proper column height designed according to mass transfer equations can also absorb nearly all the stripped ammonia (Basakilardan-Kabakci et al. 2007). As such, the recovery efficiency of ammonium in the urine would probably increase from 75% to more than 95% when operated at favourable conditions.

Precipitation of struvite and struvite-K

The precipitation of struvite is a well-known process to remove N and P from urine (Hug & Udert 2013; Ito et al. 2013; Liu et al. 2014). The precipitates were confirmed to be mainly composed of struvite in the partial scenario, corresponding to 83% removal of phosphate in the diluted urine. pH of the diluted urine (approximately 9.1) was optimal for the struvite precipitation (Ronteltap et al. 2007). Further, no significant change in the urine pH was observed after the precipitation process, although weakly acidic solution of MgCl₂ was added. As such, the phosphate removal would not be promoted with further optimisation of the solution pH. More addition of the Mg source would probably enhance the phosphate removal. Approximately 98% of the phosphate in urine was precipitated in the form of struvite at an increased Mg:P ratio of 1.5:1 (Antonini et al. 2011). However, this result apparently induces increased cost for chemical consumption.

The removal of ammonium (<3.2%) via the struvite precipitation can be ignored in the partial scenario because of more ammonium than phosphate in the diluted urine on a molar basis. Large amount of sources of Mg and P should be added according to the stoichiometric coefficient of struvite to remove most of the urine ammonium under such conditions. However, this method is not recommended because of the disadvantages of phosphate demand and inconvenience for process operation (Liu et al. 2008a, 2008b). Thus, the following process was essential considering the recovery of most of the urine ammonium.

Normally, struvite-K presents lower precipitation potential than struvite comparing their solubility products (Wilsenach et al. 2007; Xu et al. 2015). The precipitation of struvite-K was confirmed to occur only when the ammonium in the diluted urine was previously removed in the stripping column as shown in the complete scenario. The removal of K was further increased to 79% when more ammonium was stripped at an increased rate of air flow in operation III. However, approximately 20% of the urine K was still present, which did not take part in the precipitation process although the molar ratio of Mg:K:P equalled to 3:1:3 or 3.5:1:3. Herein, the precipitation of struvite-Na mainly responded for the competition with that of struvite-K for the binding with Mg²⁺ and PO₄³⁻ (Xu et al. 2015). The co-precipitation of struvite-Na weakened if less amount of NaOH was added in the stripping column. However, this effect will decrease the removal of ammonium via stripping. Therefore, the co-precipitation of struvite-Na was inevitable and more sources of Mg and P should be consumed for removing more K. The removal efficiency of K is higher than 90% when the ratio of Mg:K:P reached 6:1:6 (Xu et al. 2012). However, the cost-benefit balance between the agents added and the nutrient obtained should be considered.

Phosphate was also removed via the precipitations of struvite-K and struvite-Na. The removal efficiency was only approximately 45% at a Mg:K:P molar ratio of 3:1:3. Then, more dosing of the Mg source resulting in a Mg:K:P ratio of 3.5:1:3 again promoted the phosphate removal efficiency to 79%. The phosphate removal might be further enhanced with more addition of the Mg source. However, the removal efficiency of K would be reduced (Xu et al. 2012).

Cost and benefit issue

The electrical equipment included the air compressor, the heater, the pumps for inflow and outflow, and the pH electrodes. Energy consumption for the laboratory scale reactors in this case was not analysed because this project was not designed towards energy efficiency. A demo scale project on the stripping treatment of urine showed a high demand for energy (188 kWh m⁻³ urine) where the air compressor and heating up the liquid were the main contributors (Tettenborn et al. 2006). Maurer et al. (2006) reported that the energy consumption was approximately 7 kWh per m³
treated liquid with 95% ammonia removal at 20 °C. This result should be further studied in full project designed towards energy efficiency.

Another important contributor for the operation cost is the substance input during the reactor operations (Table 3). Both of these two operation scenarios showed no significant differences on recovering ammonium. However, the complete scenario consumed more chemicals and yielded more nutrient products for recovering K than partial scenario. Calculations showed that the ratio of substance input to nutrients recovered (in the form of P2O5 and K2O) in the complete scenario (10.61) was much higher than that in the partial scenario (4.14). This result indicates that the partial scenario is more economically efficient, whereas the complete scenario would be feasible if the K fertiliser turned out to be expensive.

CONCLUSIONS

The stripping-absorption columns were efficient in recovering ammonium from urine. The removal efficiency of ammonium increased to 99% when an increased air flow rate of 1.5 m3 h⁻¹ was employed. The absorption process in the sulphuric acid resulted in a recovery efficiency of 78% of the urine ammonium.

- Urine treatment by the precipitation reactor in advance was preferred for the recovery of the urine phosphate. Herein, approximately 81% of the phosphate was recovered mainly in the form of struvite.
- To recover most of the K, urine should be firstly treated in the stripping column to remove the ammonium to initiate the precipitation of struvite-K. The recovery efficiency of phosphate and K reached 72% and 73%, respectively, when sources of Mg and P were added to maintain a Mg:K:P ratio of 5.5:1:3. The precipitates were confirmed to be a mixture of struvite, struvite-K, and struvite-Na.
- The inevitable co-precipitation of struvite-Na resulted in the demand for sources of Mg and P to remove most of the urine K. This phenomenon led to lower economic efficiency in the complete recovery of N, P and K from urine compared with the partial scenario to recover N and P.

Table 3 | Summary of substance input and production estimated on a basis of 1 m³ urine

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<tr>
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<th>Partial scenario</th>
<th>Complete scenario</th>
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<td>MgCl₂·6H₂O (kg)</td>
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<tr>
<td>Na₃HPO₄·12H₂O (kg)</td>
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<tr>
<td>NaOH (kg)</td>
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<tr>
<td>H₂SO₄ (2M, L)</td>
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<tr>
<td>Solution of (NH₄)₂SO₄ (L)</td>
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<tr>
<td>Precipitates (kg)</td>
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<td>N (kg)</td>
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<tr>
<td>P₂O₅ (kg)</td>
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<td>K₂O (kg)</td>
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