Background and Purposes

- Nitrogen (N) pollution of ground or surface waters is a serious matter of concern in the world.
- Natural abundances of \( ^{15}N \) (\( \delta^{15}N \)) is a useful tool to estimate sources of N.
- The diffusion-based method has been used for pretreatment of \( ^{15}N \) in water samples.

- We tried to determine the best conditions for a sequential diffusion-based method to collect ammonium (NH\(_4\)-N), nitrate (NO\(_3\)-N) and total dissolved nitrogen (TDN).
- Purposes are to examine (1) necessary recovery time, (2) the range of N concentration, and (3) isotopic fractionation during the process.

Materials and Methods

We compared recovery rates of N with different concentration (inorganic N: 0-40 mg L\(^{-1}\), TDN: 0-4 mg L\(^{-1}\)) or their \( \delta^{15}N \) values (inorganic N: 20 mg L\(^{-1}\), TDN: 2 mg L\(^{-1}\)).

\[
\text{Sample name} \quad \text{Concentration (mg L}\^{-1}\text{)} \quad \delta^{15}N (\text{‰})
\]

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Concentration (mg L(^{-1}))</th>
<th>( \delta^{15}N ) (‰)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NH(_4)-N</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NH(_4)Cl (Reagent, n=3)</td>
<td>—</td>
<td>±0.9±0.0</td>
</tr>
<tr>
<td>Recovered sample (n=3)</td>
<td>20.0</td>
<td>±0.6±0.1</td>
</tr>
<tr>
<td>NO(_3)-N</td>
<td></td>
<td></td>
</tr>
<tr>
<td>KNO(_3) (Reagent, n=3)</td>
<td>—</td>
<td>−1.8±0.0</td>
</tr>
<tr>
<td>Recovered sample (n=3)</td>
<td>20.0</td>
<td>−2.5±0.1</td>
</tr>
<tr>
<td>TDN</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Glycine (Reagent, n=3)</td>
<td>—</td>
<td>±0.4±0.0</td>
</tr>
<tr>
<td>Recovered sample (n=6)</td>
<td>2.0</td>
<td>±0.2±0.4</td>
</tr>
</tbody>
</table>

\( \delta^{15}N \) values of NH\(_4\)-N, NO\(_3\)-N, and TDN recovered with PTFE traps were similar to each reagent.

Results and Discussion

N recovery rates were 100% at 0.3–20 mg L\(^{-1}\) and varied at 30–40 mg L\(^{-1}\).
In blank samples, NH\(_4\)-N concentrations were about 0.02 mg L\(^{-1}\).
→ Best range: 0.3-20 mg L\(^{-1}\).

Fig. 2 Initial vs. recovered inorganic N in low and high concentration samples.

N recovery rates were 100% at 0.2–3 mg L\(^{-1}\) and varied at 2 mg L\(^{-1}\) in low concentration samples.
In blank samples, NH\(_4\)-N concentrations were about 0.02 mg L\(^{-1}\).
→ Best range: 0.3-3 mg L\(^{-1}\).

Fig. 3 Initial vs. recovered TDN in low and high concentration samples.

Recovery rates were 100% when the amounts of N were <100 μg.
→ Limited capacity: 90 μg N of PTFE traps.

Fig. 4 Recovery rates of N for different amounts as NH\(_4\)Cl or glycine solution with concentration of 2mg L\(^{-1}\).

Conclusion

(1) Necessary recovery time:
N recovery can be shortened to 24 hours by increasing temperature to 40°C.

(2) The range of N concentration:
Inorganic N concentration should be 0.3–20 mg L\(^{-1}\) (Fig. 2).
TDN concentration should be 0.25–3 mg L\(^{-1}\) (Fig. 3) and the amount of N < 90 μg N in a vial (Fig. 4).

(3) Isotopic fractionation during the process:
No isotopic fractionation occurred during the process of this method (Table 5).