A Sequential Diffusion Method for ^{15}N Natural Abundance Measurement of Ammonium, Nitrate and Total Dissolved Nitrogen in Water Samples

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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}).

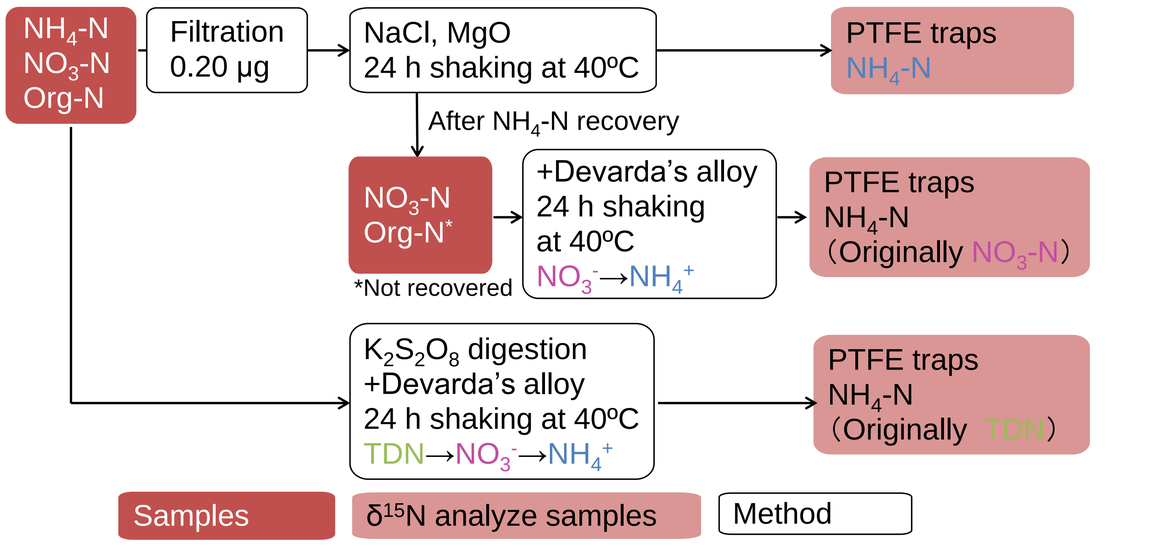

Fig. 1 Procedures to collect different forms of N.

Results and Discussion

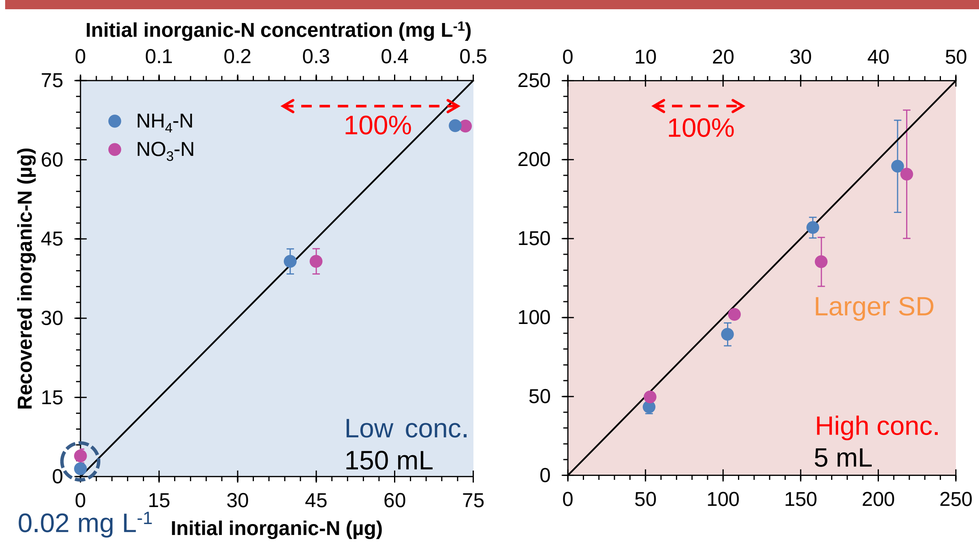

Fig. 2 Initial vs. recovered inorganic N in low and high concentration samples.

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}.

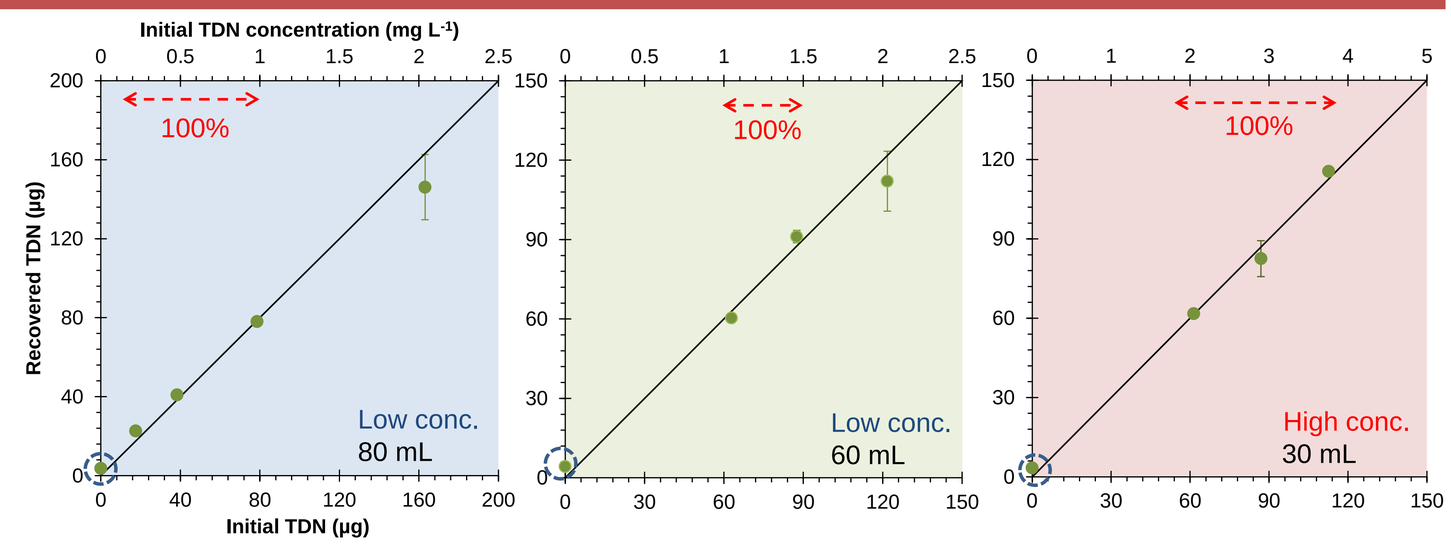

Fig. 3 Initial vs. recovered TDN in low and high concentration samples.

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}.

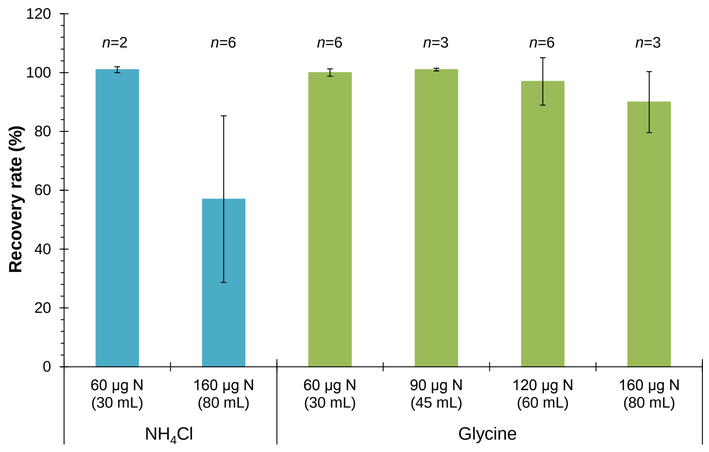

Fig. 4 Recovery rates of N for different amounts as NH_4Cl or glycine solution with concentration of 2mg L^{-1}.

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

Table 5 $\delta^{15}N$ values of recovered N by the sequential diffusion method, compared with the original chemicals.

Sample name	Concentration (mg L^{-1})	$\delta^{15}N$ (‰)
NH_4-N		
NH_4Cl (Reagent, *n*=3)	—	−0.9±0.0
Recovered sample (*n*=3)	20.0	−0.6±0.1
NO_3-N		
KNO_3 (Reagent, *n*=3)	—	−1.8±0.0
Recovered sample (*n*=3)	20.0	−2.5±0.1
TDN		
Glycine (Reagent, *n*=3)	—	+0.4±0.0
Recovered sample (*n*=6)	2.0	+0.2±0.4

—: Not measured

$\delta^{15}N$ values of NH_4-N, NO_3-N, and TDN recovered with PTFE traps were similar to each reagent.

Conclusion

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

(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).