A novel method for the extraction and measurement of hydroxylamine (NH_2OH) in soils

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1 Background

Hi! What is hydroxylamine?

It is a nitrification intermediate and involved in abiotic N_2O production[1].

Why do we want to measure it in soils?

Because it will help us to understand the role of nitrification and abiotic reactions for soil N_2O emissions.

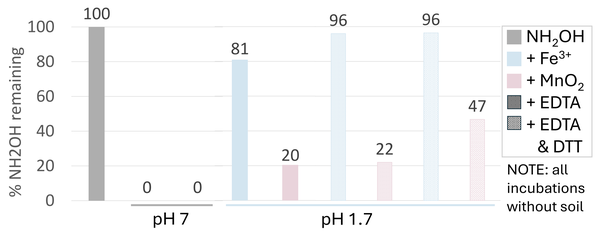
Why do we need a new method for this?

The only published extraction protocol[2] only works for a narrow range of soil types and does not achieve high recoveries.

Why is NH_2OH lost during the extraction?

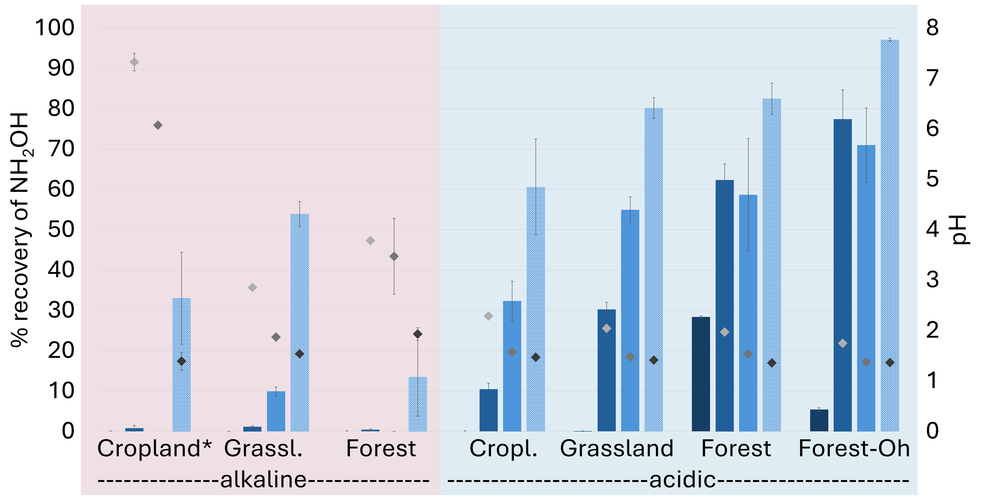
It abiotically reacts with Fe^{3+} and MnO_2 in the soil.

Can we block the reaction with Fe_3^+ and MnO_2?

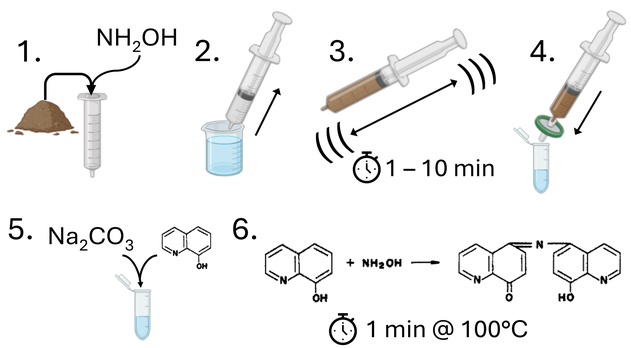


Yes, at least partially with EDTA and DTT at low pH.

3 Results: NH_2OH extraction efficiency



2 Materials & Methods



7. Measure absorbance at 705 nm

4 Conclusions

So, what do we need for a successful NH_2OH extraction?

You need a chelator (EDTA) and reducing agent (DTT), short extraction times (1min) and pH < 2 in the extract.

Is the extraction efficiency the same in all soils?

No, it is lower in $CaCO_3$-containing than in acidic soils.

To quantify soil NH_2OH contents you need to assess the extraction efficiency for each soil by spiking a known amount of NH_2OH.

5 Outlook

What will you do next?

1. Purify and concentrate the derivatization product via solid phase extraction.
2. Measure its concentration and ^{15}N-enrichment via high resolution MS.
3. Apply the method in ^{15}N-tracing studies.

References:

[1]Heil, Jannis, Harry Vereecken, and Nicolas Brüggemann. 2016. "A Review of Chemical Reactions of Nitrification Intermediates and Their Role in Nitrogen Cycling and Nitrogen Trace Gas Formation in Soil." *European Journal of Soil Science* 67 (1): 23–39.

[2] Liu, Shurong, Harry Vereecken, and Nicolas Brüggemann. 2014. "A Highly Sensitive Method for the Determination of Hydroxylamine in Soils." *Geoderma* 232–234 (November):117–22.

Derivatization reaction:
Berg, Richard, and Erna Becker. 1940. "Ein Neuer Nachweis von Hydroxylamin Durch Bildung von Chinolinchinon-(5.8)-[8-Oxy-Chinolyl 5-Imid] -(5), Genannt „Indo-Oxin"." *Berichte Der Deutschen Chemischen Gesellschaft (A and B Series)* 73 (3): 172–73.
Frear, D. Stuart, and Richard C. Burrell. 1955. "Spectrophotometric Method for Determining Hydroxylamine Reductase Activity in Higher Plants." *Analytical Chemistry* 27 (10): 1664–65.

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