# Investigating the Electrochemical Ammonia Synthesis using Chromium Nitride in a GDE-Setup

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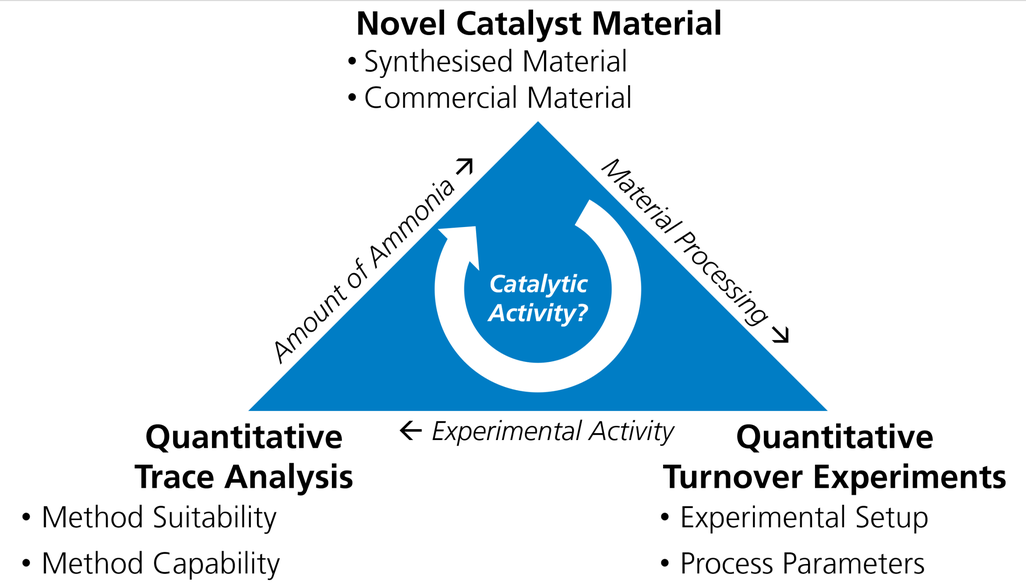
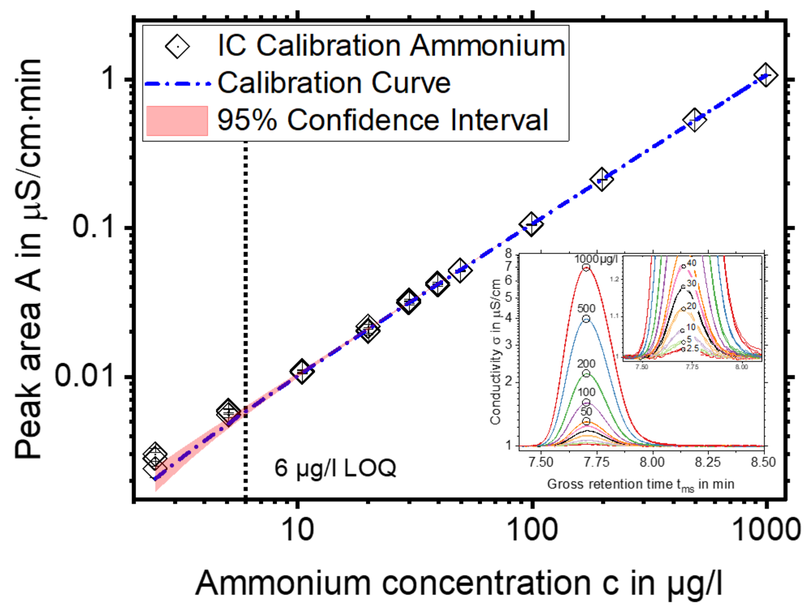
## Motivation & Approach

**Ammonia $NH_3$** is a basic chemical indispensable for today's modern agriculture and industry. The well-established $CO_2$-intensive **Haber-Bosch process** used for the industrial scale production of ammonia on the order of 100 Mt/a is ill-suited for a scale down and production at point of use. The emerging **Electrochemical Ammonia Synthesis (EAS)** is of great recent interest for this prospective scalable **green ammonia production**. However, low experimental production rates so far require **novel** highly active and selective **electrocatalysts**. Assessing catalyst for the EAS entails challenging quantitative **trace level determination of ammonia** in experimental conditions. Herein, we show the application of **Ion Chromatography (IC)** to experimental **Gas Diffusion Electrode (GDE)** measurements of **Chromium Nitride (CrN)** as theoretically promising electrocatalyst for EAS.




## Trace Level Determination of Ammonium

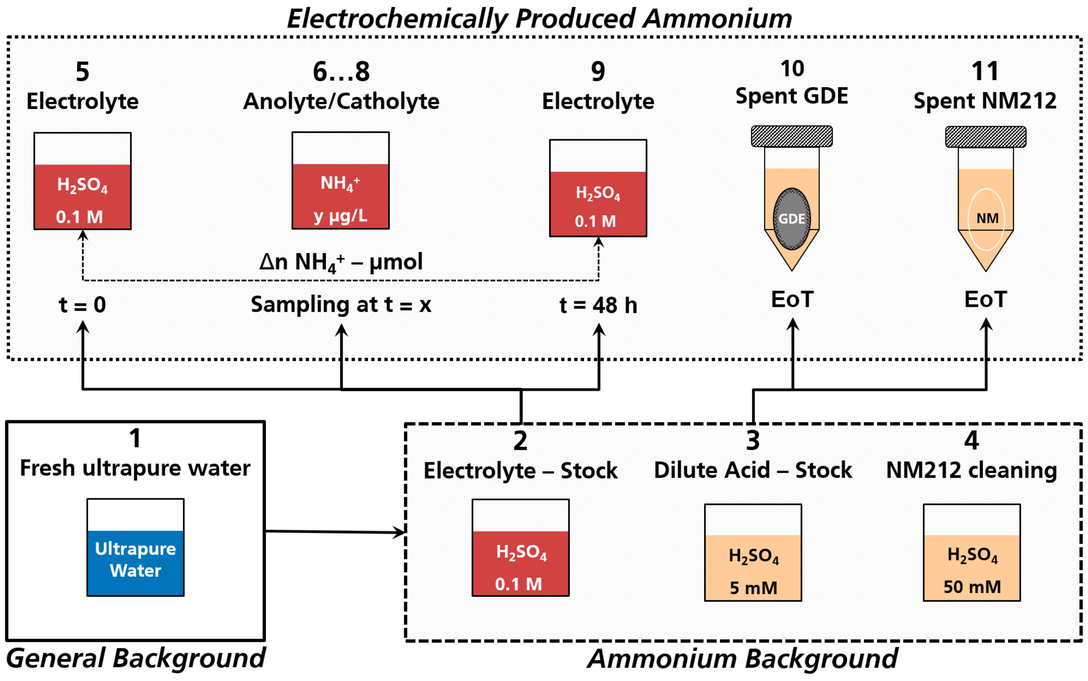
Trace level determination of ammonium via **ion chromatography** (**IC**) using a high-capacity weak-acid **separation column Metrosep C6 250/4.0**




Ion chromatography calibration for ammonium from 2.5 to 1000 µg/l measured in triplicate by unsuppressed conductivity detection

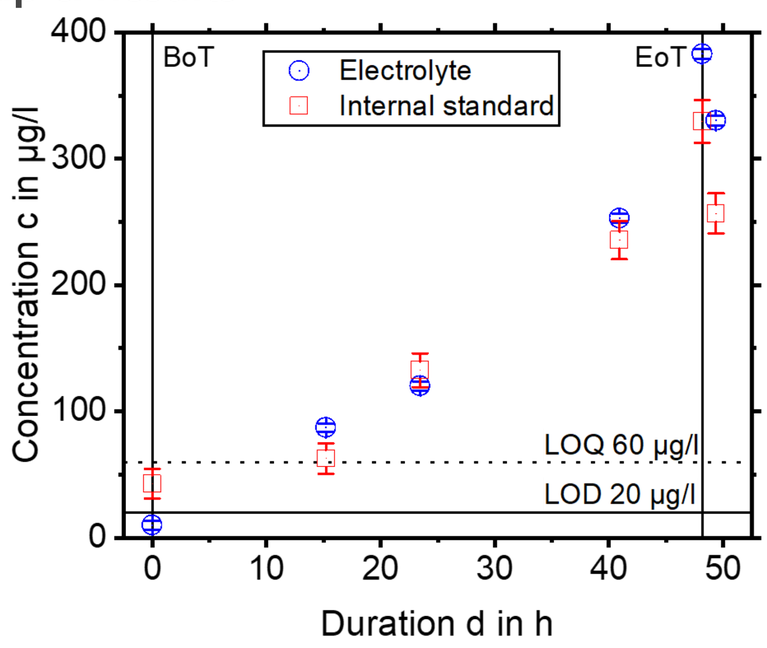
- Achieving a limit of quantification (**LOQ**) of **6 µg/l** (**ppb**) ammonium!
- Fast analysis time of 20 min – **Automated processing** by autosampler
- Well calibrated IC → Fast, sensitive, automated quantitative analysis

Extensive **sampling** and routine analysis of **stock solutions** for **ammonium background** correction in experiments and possible contamination




Stock solutions and derived samples for background correction to resolve the time evolution of the ammonium concentration in real-world EAS experiments

## Setup & Results




**Setup**

- **Electrode**: GDE with MPL Freudenberg H2315 C6 I2, target loading of 1 mg/cm² catalyst, 30 w% Nafion in coating, Ø28 mm, ionomer membrane NM212 pre-cleaned and hot-pressed
- **Setup**: Gaskatel FlexCell® measurement cell, electrolyte 0.1 M $H_2SO_4$, room temperature, dry nitrogen 30 ml/min, potential -0.2 V vs. RHE, 48 h
- **IC-Analytic**: Samples of 0.5 ml electrolyte collected at different intervals, topped up with stock electrolyte, diluted 1:9 with 0.011 M KOH for analysis

**Results**

- The used **quantitative trace analysis** of ammonium and used **GDE-setup proved suitable** for EAS experiments
- The derived production rate of **1.67±0.1 pmol/s/cm²$_{geo}$** is **comparable to literature** results of a membrane electrode assembly (MEA) setup at 80 °C [1]

## Outlook

- Investigate reproducibility and membrane impact with further measurements and in different configuration (H-cell)
- Extend to current state of the art protocol by dedicated background measurements at open circuit potential (OCP) with nitrogen and argon

## References

[1] J. Nash, et al., *Elucidation of the Active Phase and Deactivation Mechanisms of Chromium Nitride in the Electrochemical Nitrogen Reduction Reaction.* The Journal of Physical Chemistry C, 2019. **123** (39): p. 23967-23975.

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