Changing the analysis method during the long-term monitoring project - TN analysis with TOC/TN

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The research chain

Planning

Results evaluation

Publications
Why changes in the laboratory?

Resources:

Staff
Financial resources
Organisation changes

Laboratory:

User-friendly methods
Cost-effectiveness
Data system or software changes
Why changes in the laboratory?

**Device:**
- Analytics develop
- The old device breaks
- Knowledge

**Quality system:**
- Accreditation
- Validation of the method
  - UC, LOQ
Determination of the total (Dissolved) Nitrogen

Flow Injection Analyser
(peroxodisulfate/120°C + Cd column reduction)

\[
\text{NH}_4^+ + \text{NO}_3^- + \text{NO}_2^- + \text{N}_{\text{org}} \rightarrow \text{NO}_3^- \rightarrow \text{NO}_2^-
\]

**UC:** ? **LOQ:** 50 µg/l
Determination of the total (Dissolved) Nitrogen

TOC-TN-analyser/NPOC
(catalytic combustion/720°C/Pt catalyst + chemiluminescence detection NDIR)

\[ 2 \text{NO}_2^- + \text{Pt Catalyst} \rightarrow 2 \text{NO} + \text{O}_2 \]

**UC:** 18%  **LOQ:** 60 µg/l
DN method with $\text{TOC-L}_{\text{CSH/TNM-L}}$

DOC (NPOC) and DN from the same sample with TOC/TN catalyst

1) $\text{TOC/DOC} = \text{NPOC}$ \quad (TOC = TC − IC)
   pH2-3, bubbling $\rightarrow$ detection of DOC
2) DN measurement

DN analyses with and without bubbling were tested $\rightarrow$ ok
TOC/TN-analyser/DN vs. FIA/DN

Overall validation of the TOC/TN methods and they were approved by FINAS (2012)
- soil solution and deposition sample results were compared with FIA (2012)
- deposition samples (open field and throughfall) were compared in more detail (2013)

- No difference between the soil solution and deposition samples, no trends
TOC/TN-analyser/DN vs. FIA/DN

FIA DN results were ca 8 % higher compared to DOC/DN
→ within uncertainty

Differences between the methods?
➢ Digestion process/analysis
➢ Systematic error of the method
TOC/TN-analyser/DN vs. FIA/DN

Mean values in X control charts

FIA 1%

1.8 % TOC/TN

The control sample values of the both methods are within the warning limits
Conclusions

TN analysis of the soil and deposition samples are analysed with TOC/TN analyser

- Limits of quantification are sufficient
- Accuracy
- User-friendly method
- Cost-effectiveness

the FIA method

- Limits of quantification are low (ca. 30 µg/l)
- Accuracy
Thank you