



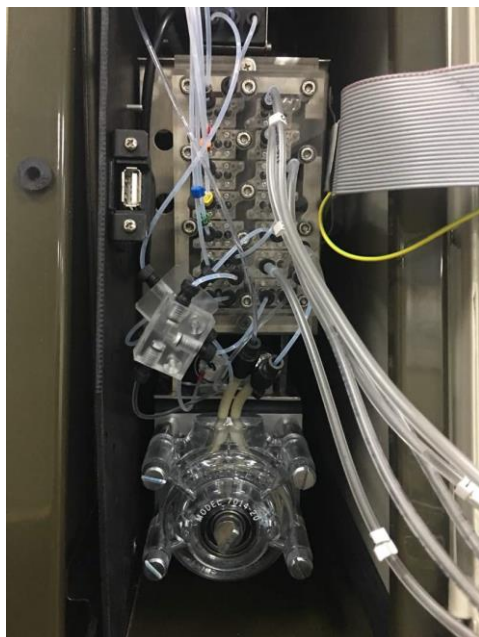
Field test of a reliable and easy to use microLFR based nutrient sensor package for Ferrybox on-line monitoring applications

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Micromac-1000 Total P & NH₃ with μ LFR hydraulics (2017)



New features:

- ❖ Teflon sealed hydraulics
- ❖ Lower reagents consumption enables longer unattended operation
- ❖ Smaller reagents volume -> internal reagents cooling for longer unattended operation



Trans National Action, Jerico-NEXT project Easy On-Line microLFA

Field long term test of
n.3 biparametric sequential Micromac-1000
on-line analyzers:

1. NH_3 (fluo) and PO_4 (fluo)
2. NO_2 and NO_3+NO_2
3. PO_4 and SiO_2

on board of Color Fantasy Ferrybox

Field test
supported by:





Nutrient instrumental configuration for Ferrybox systems

Micromac-1000 NH₃ & PO₄

- ❖ NH₃ by OPA fluorimetric method, cal. range 0-100 µg/L as N, MDL < 1.2 µg/L, measurement time (mt) 15 minutes
- ❖ PO₄ by Rhodamine 6G + Molybdate fluor. method, cal. range 0-100 µg/L as P, MDL < 3.5 µg/L, mt = 21 min

Micromac-1000 MP2 NO₂ & NO_x

- ❖ NO₂ by NED-SAA colorimetric method, range 0-50 µg/L as N, MDL < 0.4 µg/L, mt = 13 min
- ❖ NO₂+NO₃ by VCl₃ red. and NED-SAA col. method, cal. range 0-350 µg/L as N, MDL < 2.5 µg/L, mt = 17 min

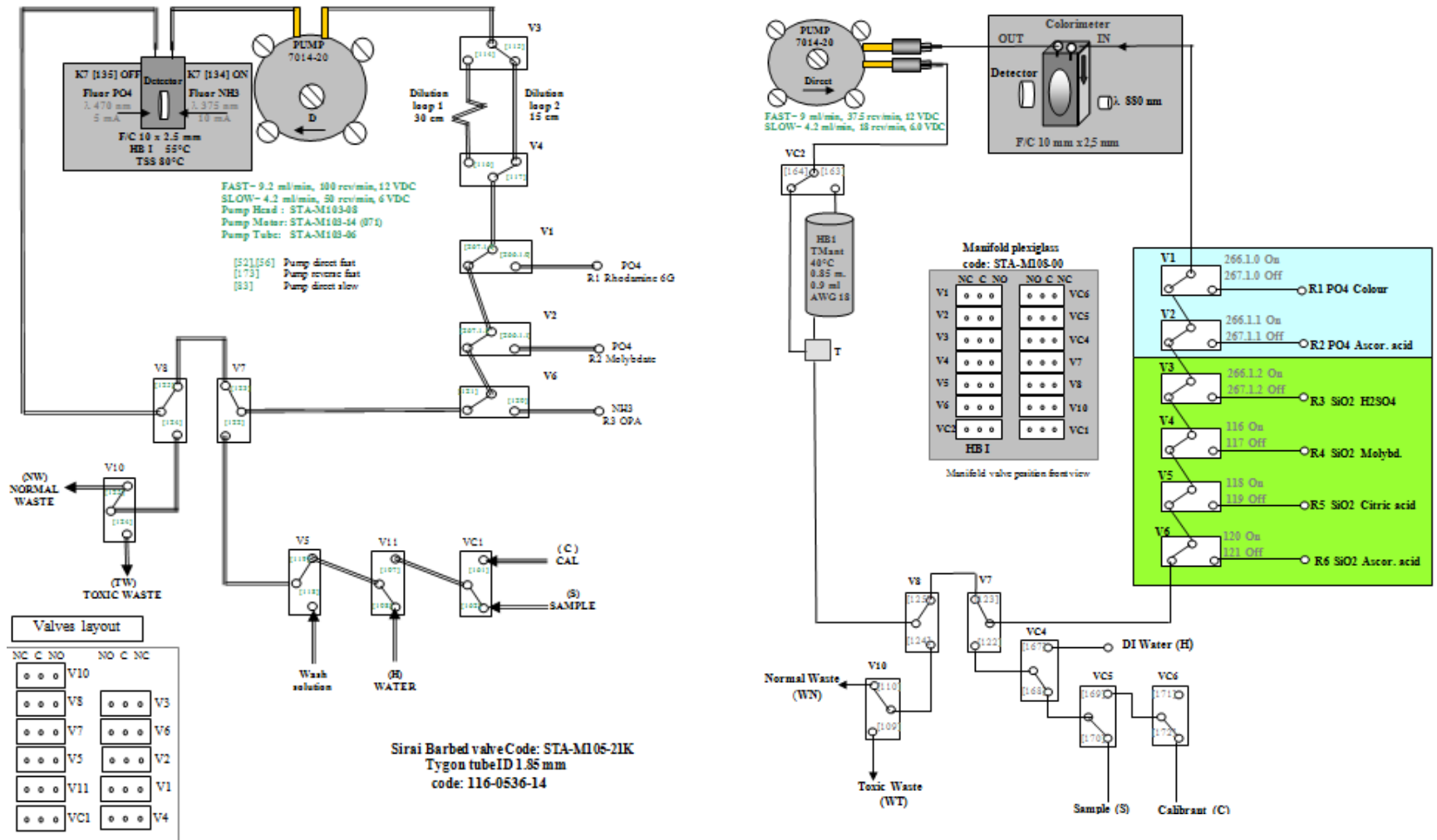
Micromac-1000 MP2 PO₄ & SiO₂

- ❖ PO₄ by Molybdenum blue colorimetric method, cal. range 0-100 µg/L as P, MDL < 1.1 µg/L, mt = 15 min
- ❖ SiO₂ by Meta-Molybdenum col. method, cal. range 0-300 µg/L as SiO₂, MDL < 3.5 µg/L = 21 min

MDL calculated as 3 * calculated std dev on 7 repetitions at 10% F.S.



μLoop Flow Reactor hydraulic diagrams



NH₃ & PO₄ F

PO₄ & SiO₂



Reagents consumption for each analysis

Parameter	Reagent 1/3 (µL)	Reagent 2/4 (µL)	DI water (mL)
NH ₃	70	70	25
PO ₄ fluo	140 70	140 70	25
NO ₂	70	70	10
NO _x	140 280	140	10
PO ₄	70	70	25
SiO ₂	140 70	140 70	25



Ferrybox installation

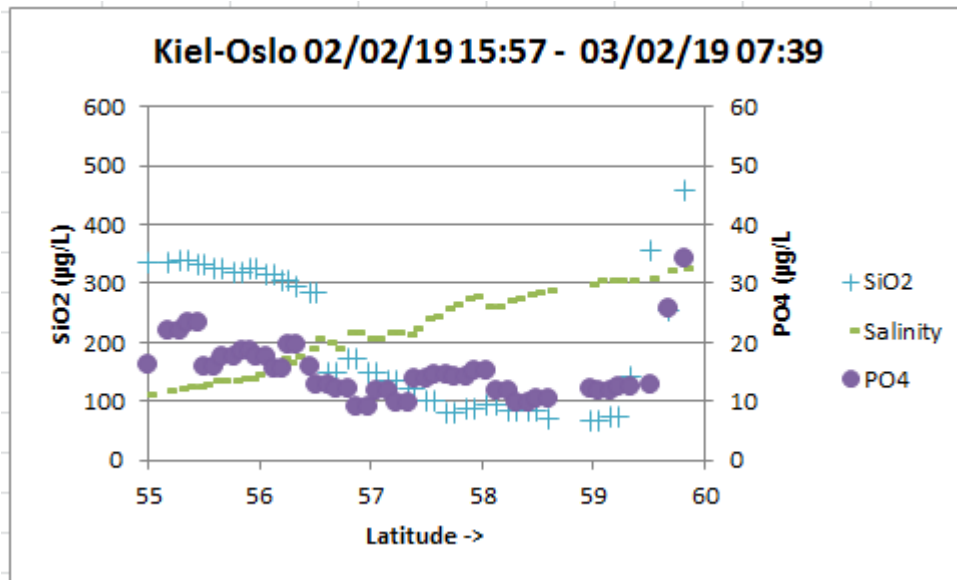
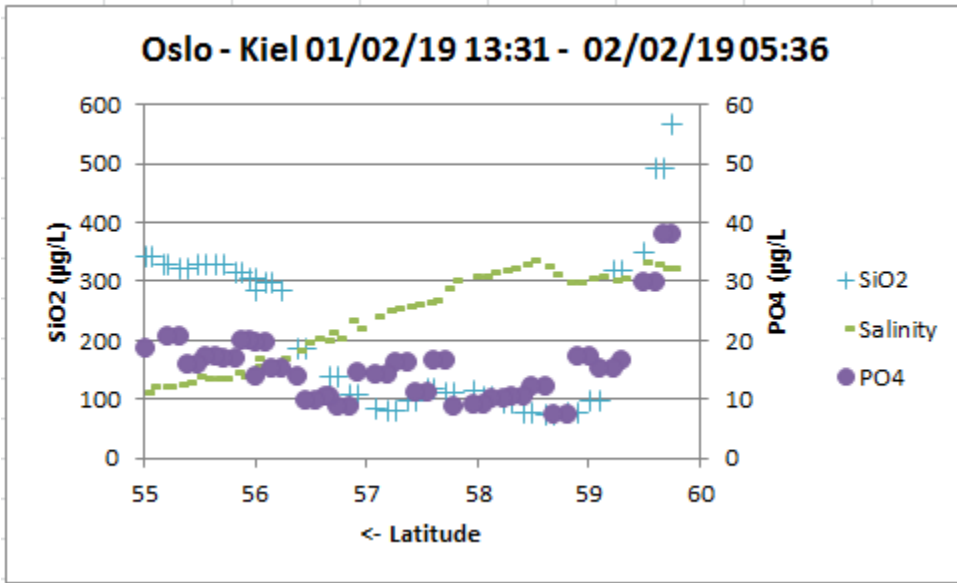


Ferrybox operations

- Reagents and distilled water refilled approximately ~1-2x per month
- Data saved from Micromacs to FerryBox PC and correlated with GPS data + temperature and salinity
- Data transferred via satellite internet to FTP server at NIVA
- 27 discrete nutrient samples collected on 26-27 February and 8-10 April 2019 for lab analysis comparison

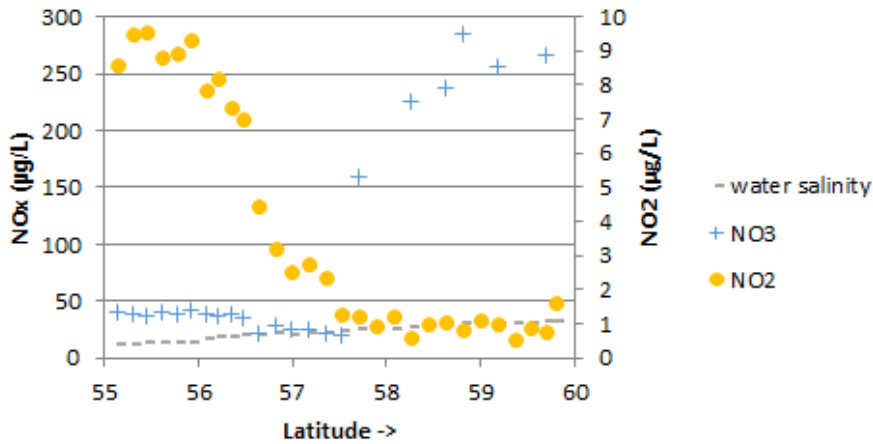


PO₄ & SiO₂ first collected data

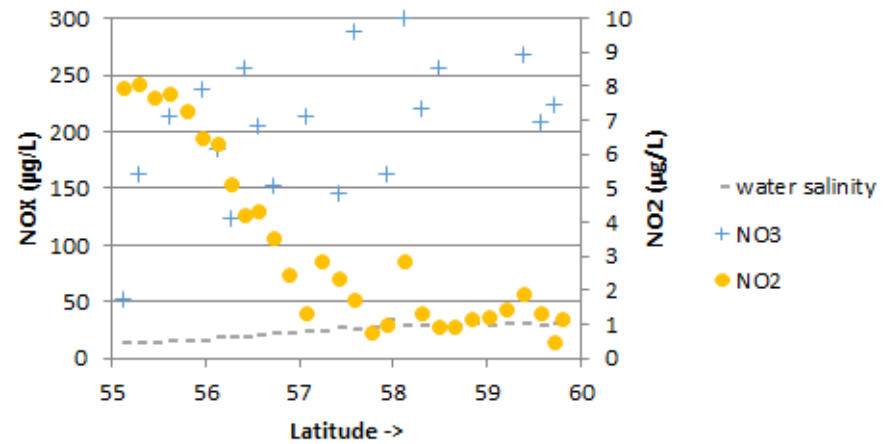


NO₂ trend repeatability along consecutive Kiel -> Oslo trips

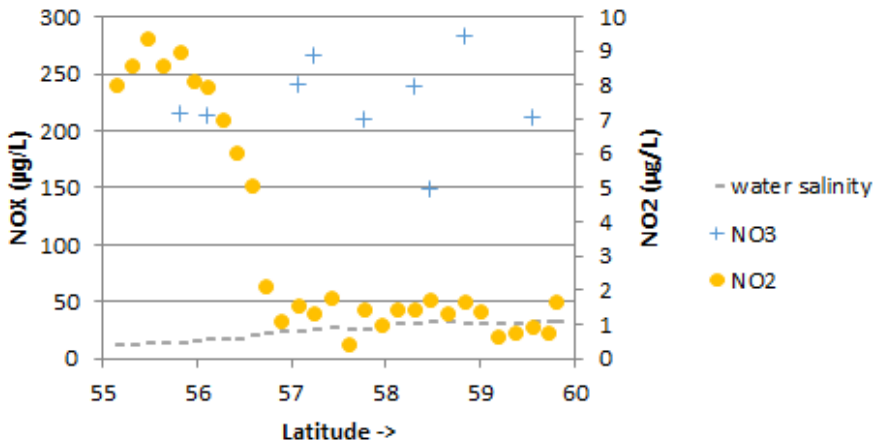
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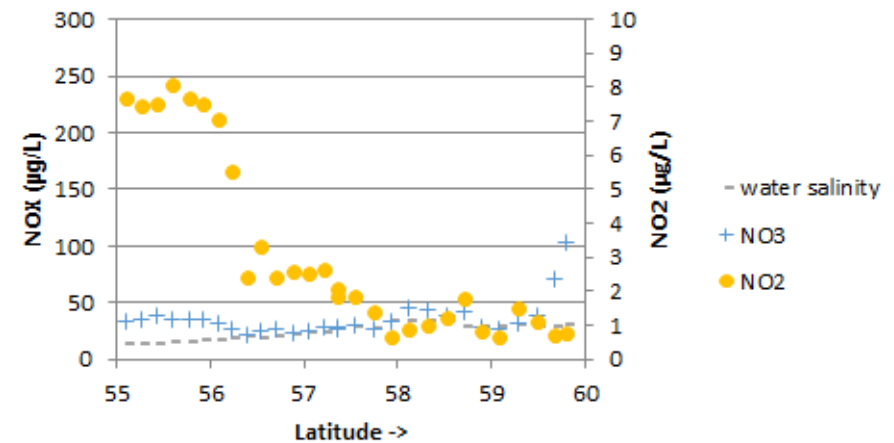
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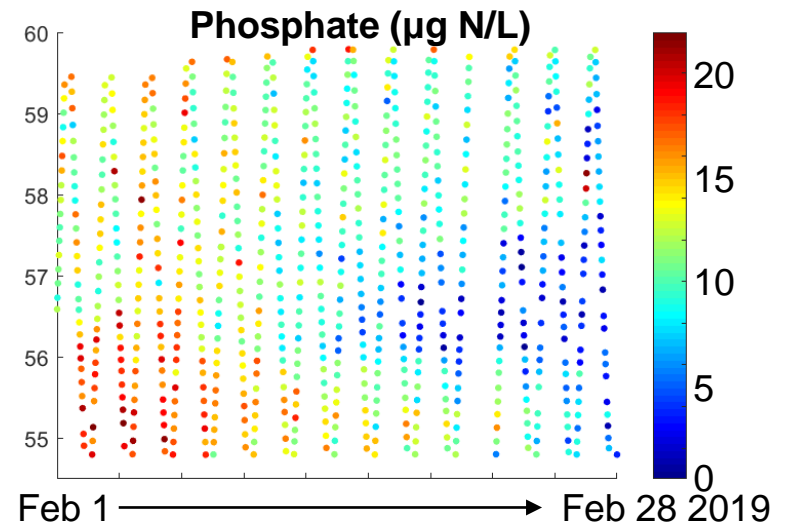
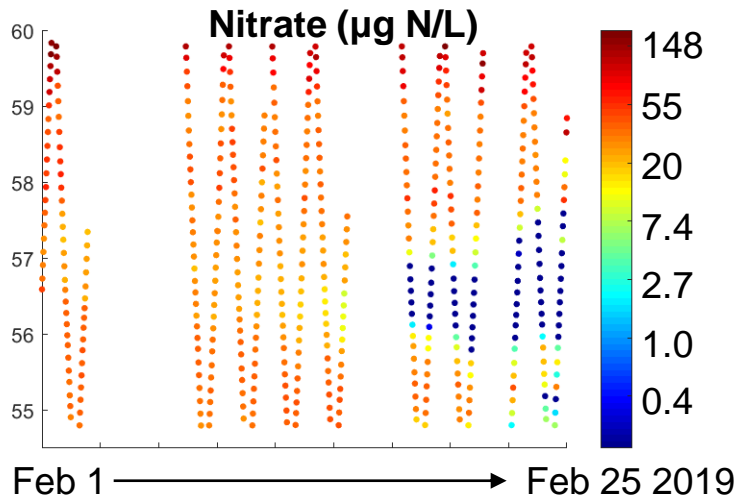
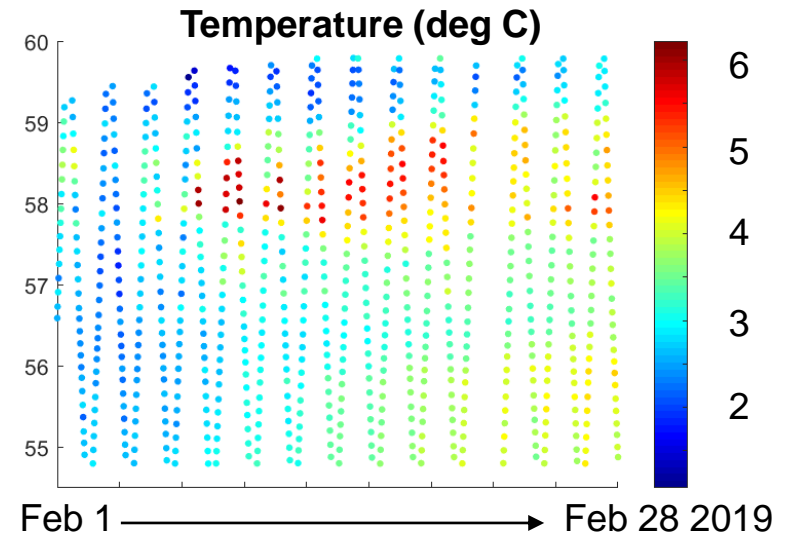
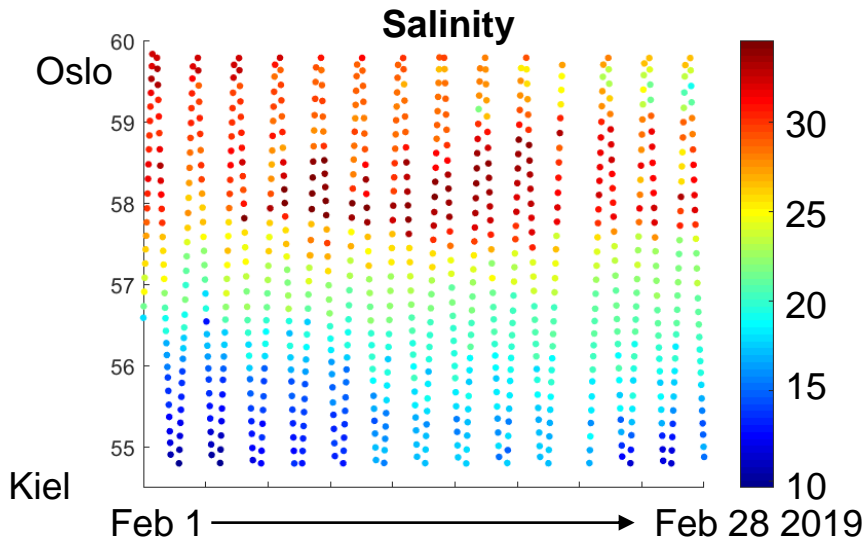
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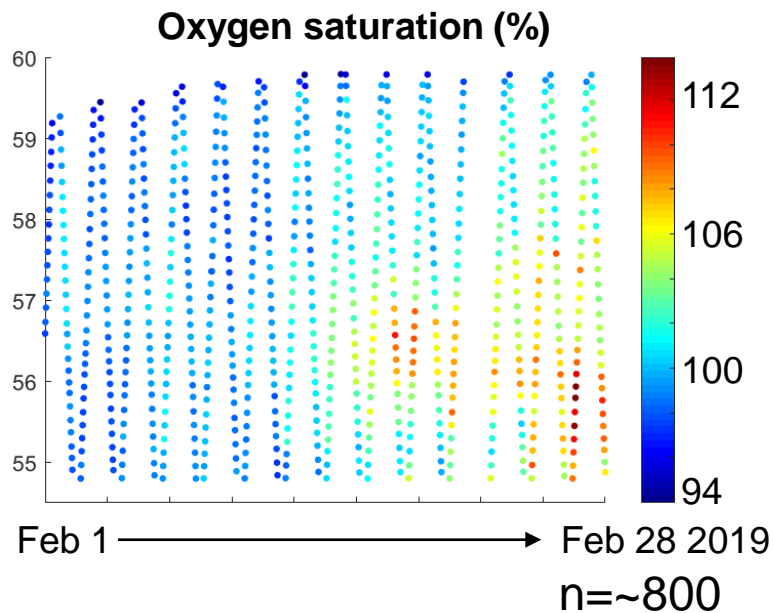
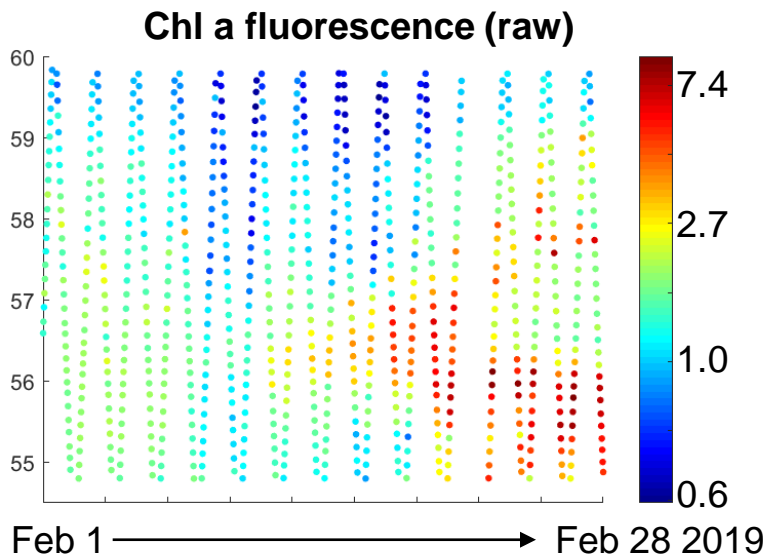
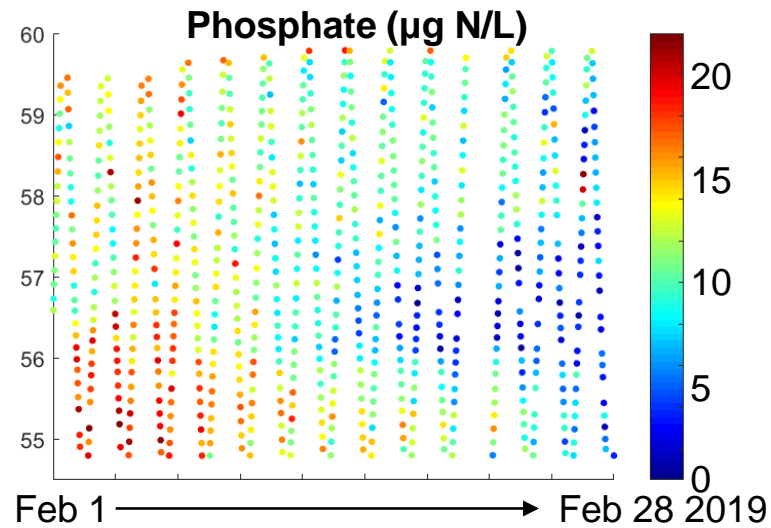
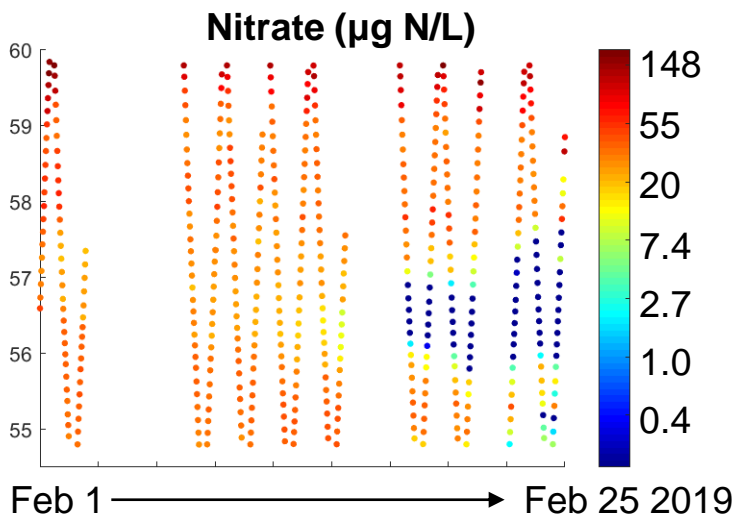


Nutrients in relation to S and T



n= \sim 800

Nutrients in relation to chl a



Positive control check after 2.5 months of operation*

Parameter	Standard (µg/L)	Value (µg/L)	% change
NH ₃	100	80	-20%
NO ₂	50	47.5	-5%
NO ₃	350	260	-25,7%
PO ₄	100	82	-18%
SiO ₂	300	--	--

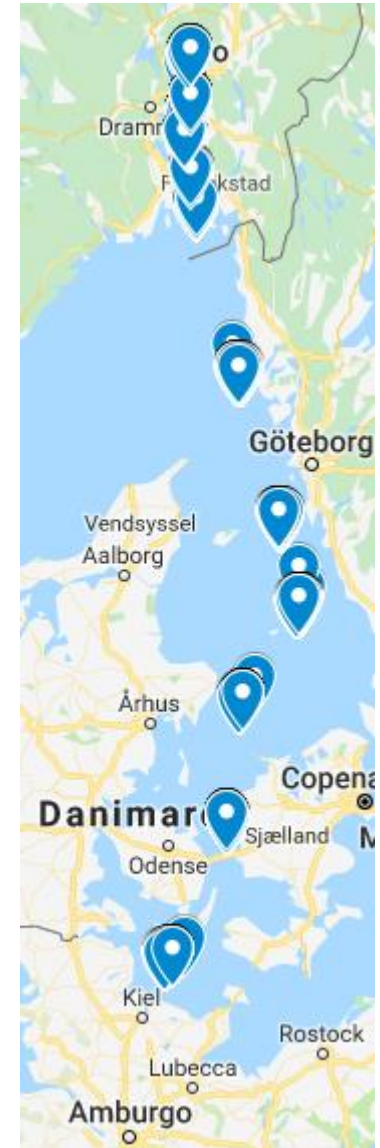
* Without any recalibration
 Reagents refilled 7 March
 Control check on 12 April



Water samplings and laboratory measurements

- Some of the samples were collected manually during the trip and preserved directly adding 1 mL H₂SO₄/100 mL of water sample
- When automatic sampling were done, water samples were preserved in Oslo harbour
- Preserved samples were measured in lab all together using a Skalar CFA analyzer on 15 April.

Anyway, no cross check measurements on the collected water samples were done using Micromac analyzers.



Issue	Action done
Data storage every minutes didn't allow an easy and direct data interpretation and evaluation	Data filtration by Excel macro. NIVA's data-logger protocol update to store only real measured data
SiO ₂ missing data after 15 days of operation, due to the use of an alternative reagent generated crystals blocking reagent and sampling line	Use of the right reagent as requested by the standard method
PO ₄ fluo collected too high concentrations due to missing DI water generated false ODS values	DI water consumption to be minimized by measurement cycle update and internal control check
Missing correlation between PO ₄ fluo and PO ₄ colo data	Planned on board activity in 2 nd week of May to cross check the issue
NOx too high values over some periods	Planned on board activity in 2 nd week of May to cross check the issue

Issue	Action done
Missing automatic positive controls even if on board standards were available	SW improvement in NIVA data-logger to perform automatic positive controls along the trip
Missing periodic recalibration of the analyzers	To be done manually in harbour based on results of positive control checks
Missing written procedure to perform on board maintenance	Written procedure was defined and started to be tested on board
Difficulty to correlate GPS position of collected water samples with on-line nutrients data, due to continuous sample flow in the Ferrybox	Installation of a storage tank to be updated periodically, where the same sample from Micromac and lab water sample have to be collected
Difficulty of cross comparison between lab and on-line measurements	Lab samples will be measured again on Micromac analyzers, when the Ferrybox is stopped in Oslo using the grab sample function.

Conclusions

- Easy installation and start-up was performed on site in two days
- 2,5 months of acquired unattended data with very good reagents stability, even if some issues were reported on collected data
- Planned joint on board activity to solve the PO_4 fluo and NO_x pending issues on 2nd week of May
- Automatic positive controls have to be activated by NIVA data-logger, to support on board validation of the collected data
- Collected water samples will have to be measured on Micromac analyzers when the Ferrybox will be stopped in Oslo
- Field test will go on further until end of June 2019
- User manual and technical training to be improved, to allow a reliable independent use from the very beginning
- Strict management procedure is required to manage properly long term unattended analysis.