

Cost effective sensing platform for the detection of phosphate in natural waters

Andrew Donohoe, Margaret McCaul, Gareth Lacour,
Dermot Diamond



Ireland's European Structural and
Investment Funds Programmes
2014-2020

Co-funded by the Irish Government
and the European Union



European Union
European Regional
Development Fund



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Insight Centre for Data Analytics



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MINISTER BRUTON LAUNCHES €88 MILLION SFI RESEARCH CENTRE, BRINGING NEW INSIGHTS TO DATA ANALYTICS

- Insight, the Centre for Data Analytics, will position Ireland at the heart of global Data Analytics research
- The largest investment in a single research centre in the history of the state

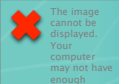
‘Insight Centre for Data Analytics’

- Uniting universities, 40 industry partners and 200 researchers in the multi-location research centre
- Creating 300 direct jobs through 12 funded spin outs, as well as creating indirectly thousands of other job opportunities

12th December 2013: The Minister for Jobs, Enterprise and Innovation, Mr Richard Bruton T.D. and Minister for Education and Skills, Mr. Ruairi Ó Riada, today officially launched the new multi-location Science Foundation Ireland (SFI) Research Centre for Data Analytics. In a joint initiative between DCU, NUI Galway, UCC and UCD, Insight, and other partner institutions, brings together more than 200 researchers from these and other Higher Education institutions, with 30 industry partners, to position Ireland at the heart of global data analytics research.

The Centre will receive funding of €58 million from the Department of Jobs, Enterprise and Innovation through SFI's Research Centres Programme, along with a further contribution of €30 million from 30 industry partners. Insight represents a new approach to research and development in Ireland, by connecting the scientific research of Ireland's leading data analytics researchers with the needs of industry and the enterprise.

- Biggest single research investment ever by Science Foundation Ireland
- Biggest coordinated research programme in the history of the state
- Focused on ‘big data’



OÉ Gaillimh
NUI Galway





Principal Investigator - Professor Dermot Diamond

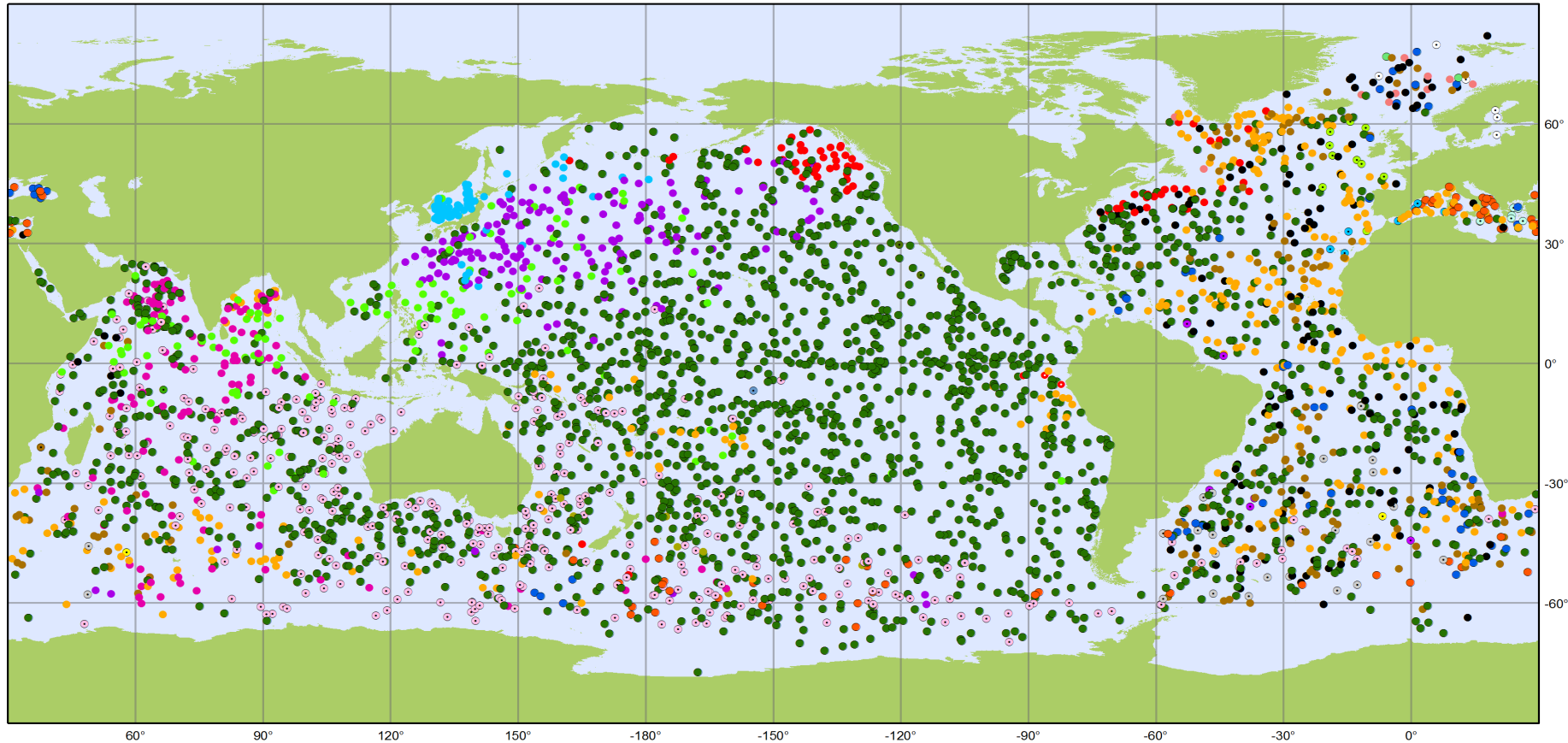
Create cost effective sensors that can obtain accurate, real-time information about environmental status (mainly related to water quality) from the highly local to global scale.

This can only be realised through ‘deploy and forget’ models of use, in which the analytical platforms are:

- i) capable of autonomous function for periods of months between servicing intervals;**
- ii) provide validated analytical data over this period,**
- iii) are relatively inexpensive to buy and maintain**



Argo project – National Contributions



Argo

National contributions - 3904 Operational Floats

May 2017

Latest location of operational floats (data distributed within the last 30 days)

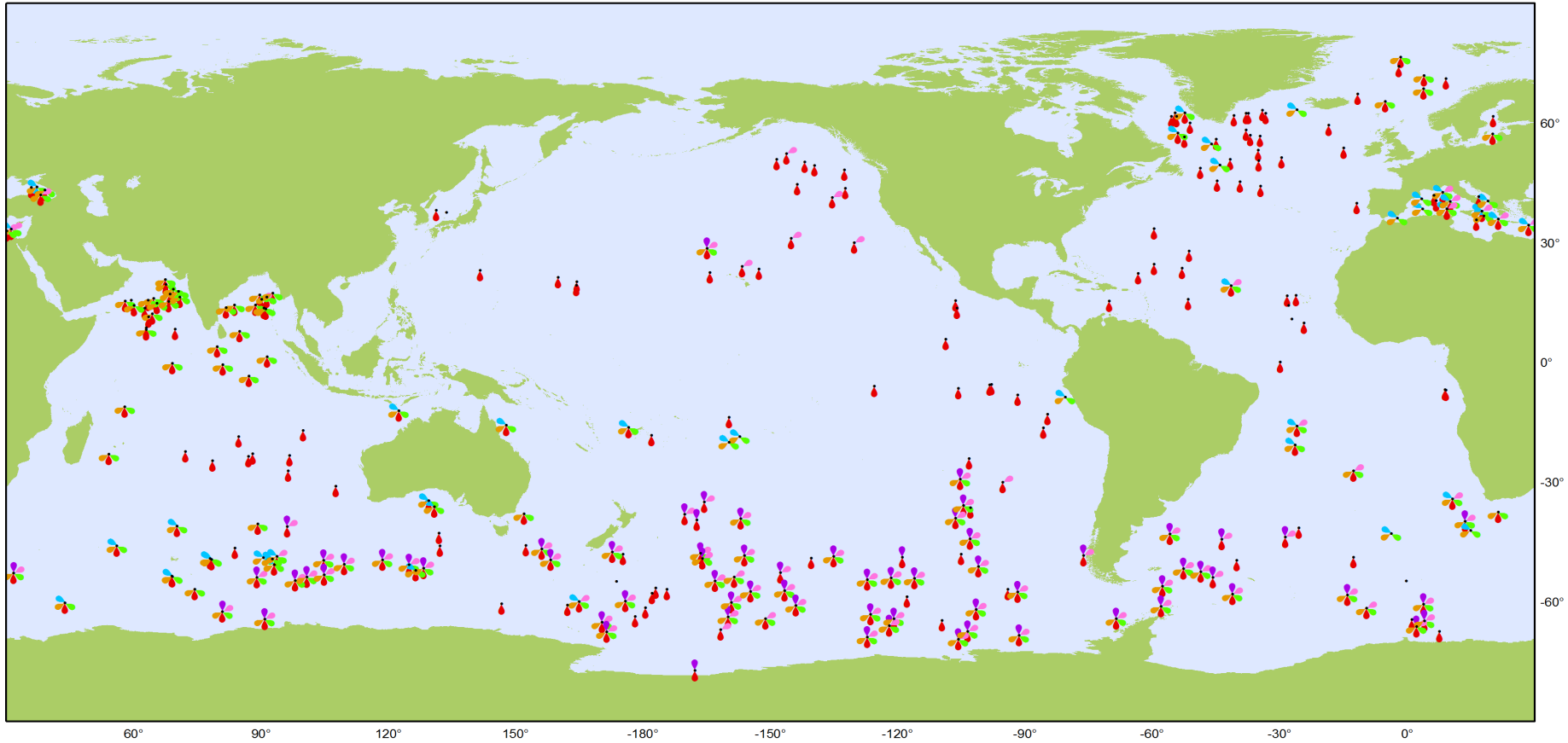
- | | | | | | |
|-------------------|-----------------|----------------|--------------------|---------------------------|--------------|
| ● ARGENTINA (2) | ● ECUADOR (1) | ● GREECE (8) | ● KENYA (1) | ● NORWAY (10) | ● UK (156) |
| ● AUSTRALIA (370) | ● EUROPE (59) | ● INDIA (130) | ● MAURITIUS (1) | ● PERU (3) | ● USA (2142) |
| ● BRAZIL (6) | ● FINLAND (6) | ● IRELAND (10) | ● MEXICO (2) | ● POLAND (2) | |
| ● CANADA (74) | ● FRANCE (319) | ● ITALY (70) | ● NETHERLANDS (26) | ● KOREA, REPUBLIC OF (62) | |
| ● CHINA (116) | ● GERMANY (143) | ● JAPAN (171) | ● NEW ZEALAND (7) | ● SPAIN (7) | |



Accessed 19 June 2017 ; <http://www.jcommops.org/board?t=Argo>

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Argo project – Biogeochemical Sensors



Biogeochemical Argo

Sensor Types

May 2017

Latest location of operational floats (data distributed within the last 30 days)

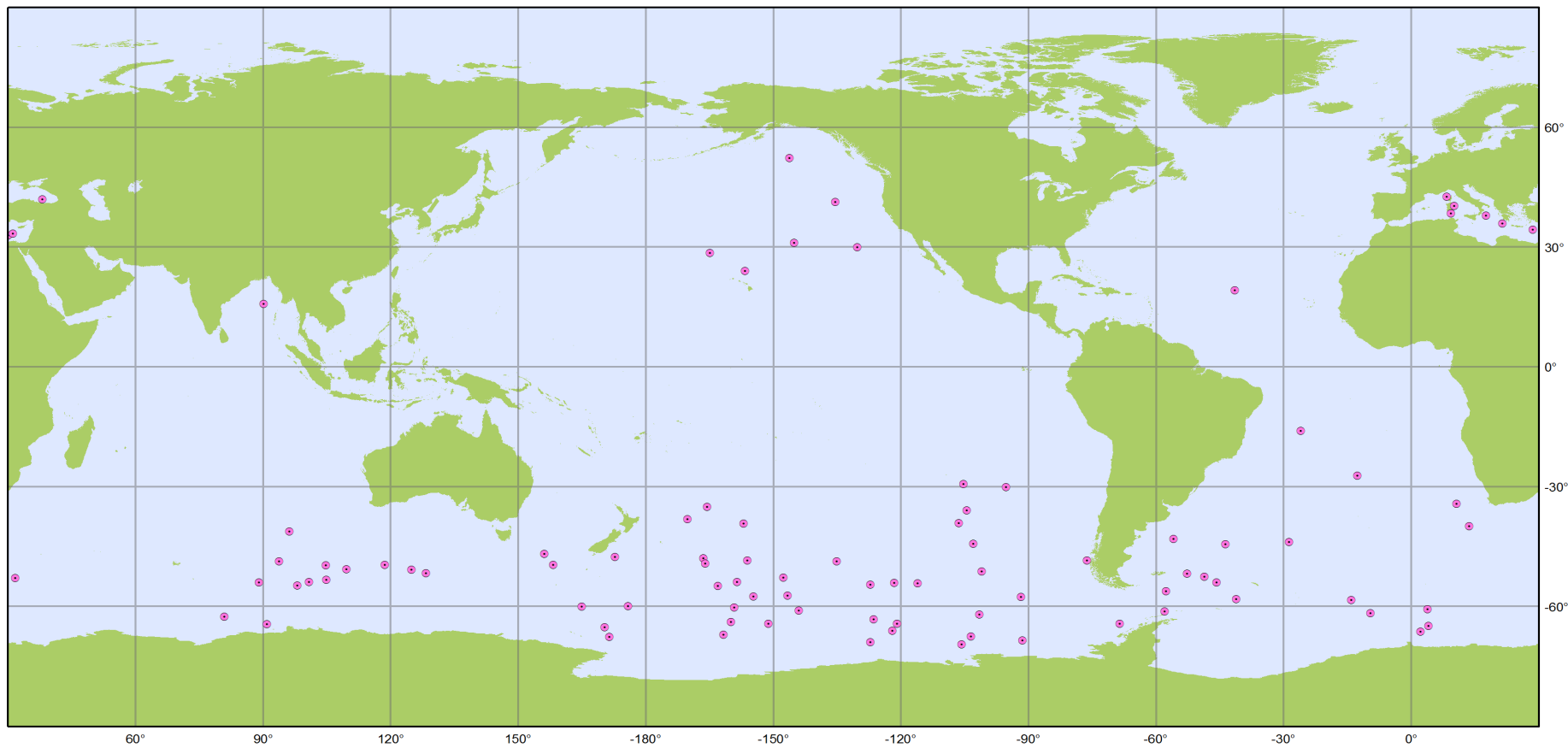
- Operational Floats (284)
- Suspended particles (149)
- Downwelling irradiance (47)
- pH (70)
- Nitrate (93)
- Chlorophyll a (150)
- Oxygen (264)



Accessed 19 June 2017; <http://www.jcommops.org/board?t=Argo>

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Argo project – Nitrate Sensing



Argo

BioGeoChemical Argo - Nitrate

May 2017

Latest location of operational floats (data distributed within the last 30 days)

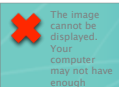
Cost €60,000 per Sensor

● SPECTROPHOTOMETER_NITRATE/BISULFIDE (93)



Accessed 19 June 2017 ; <http://www.jcommops.org/board?t=Argo>

Generated by www.jcommops.org, 02/06/2017





COMMON SENSE
marine sensors - marine monitoring



Title: Cost-effective sensors, interoperable with international existing ocean observing systems, to meet EU policies requirements

Total Budget: €6,074,497

Duration: 40 months

Consortium: 15 partners from seven different countries

(the COMMON SENSE consortium comprises six SMEs, five research development institutes, three universities and one foundation)



Common Sense - Aims

Develop innovative, cost-effective sensors that will increase the availability of standardised data on:

- **Eutrophication** DCU Responsible for Nutrient Detection
- Concentrations of heavy metals;
- Micro-plastic fraction within marine litter;
- Underwater noise
- Parameters such as temperature and pressure.

Sensors will assess environmental conditions affecting marine ecosystems:

- Mitigating the anthropogenic impacts
- Climate change impacts
- Promoting basic research of marine science



European Legislation

COMMONSENSE and the Marine Framework Directive

Under the **Marine Strategy Framework Directive (MSFD)**, EU Member States are expected to assess the overall status of their marine environments and to put in place the necessary measures to achieve Good Environmental Status (GES) by 2020. Member States must implement cost-effective monitoring programmes in order to achieve MSFD monitoring objectives, as well as other European maritime and environmental policies such as the Common Fisheries Policy (CFP).



http://science.gu.se/digitalAssets/1322/1322948_nodularia-bloomning_460px.jpg



Sensor Development

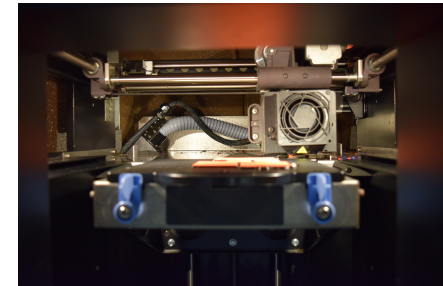


Develop Sensors for nutrients (nitrite, nitrate and phosphate) based on:

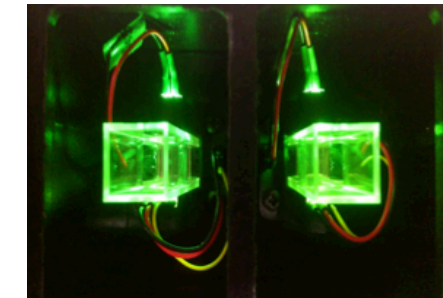
- **Colorimetric chemical assays**
- **Rapid Fabrication and Prototyping**
- **Fluidic Control**
- **LED Based Microfluidic systems**
- **Wireless Communications**



Colorimetric Detection of Phosphate



In-house 3D printing



LED Based Detection

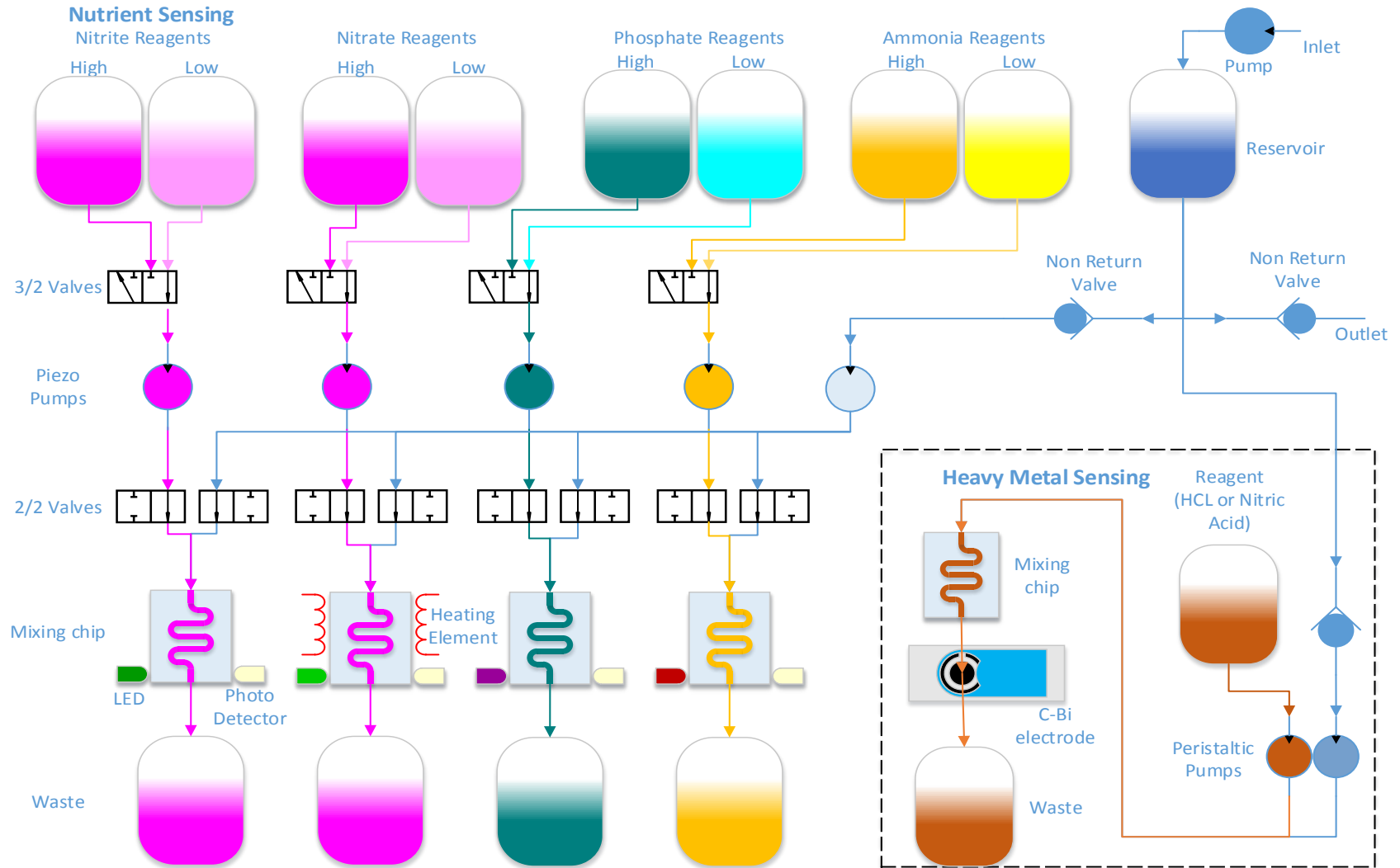
Prior to Integration

- **Individual Component testing and Validation**
- **Validation of Colorimetric Assays**



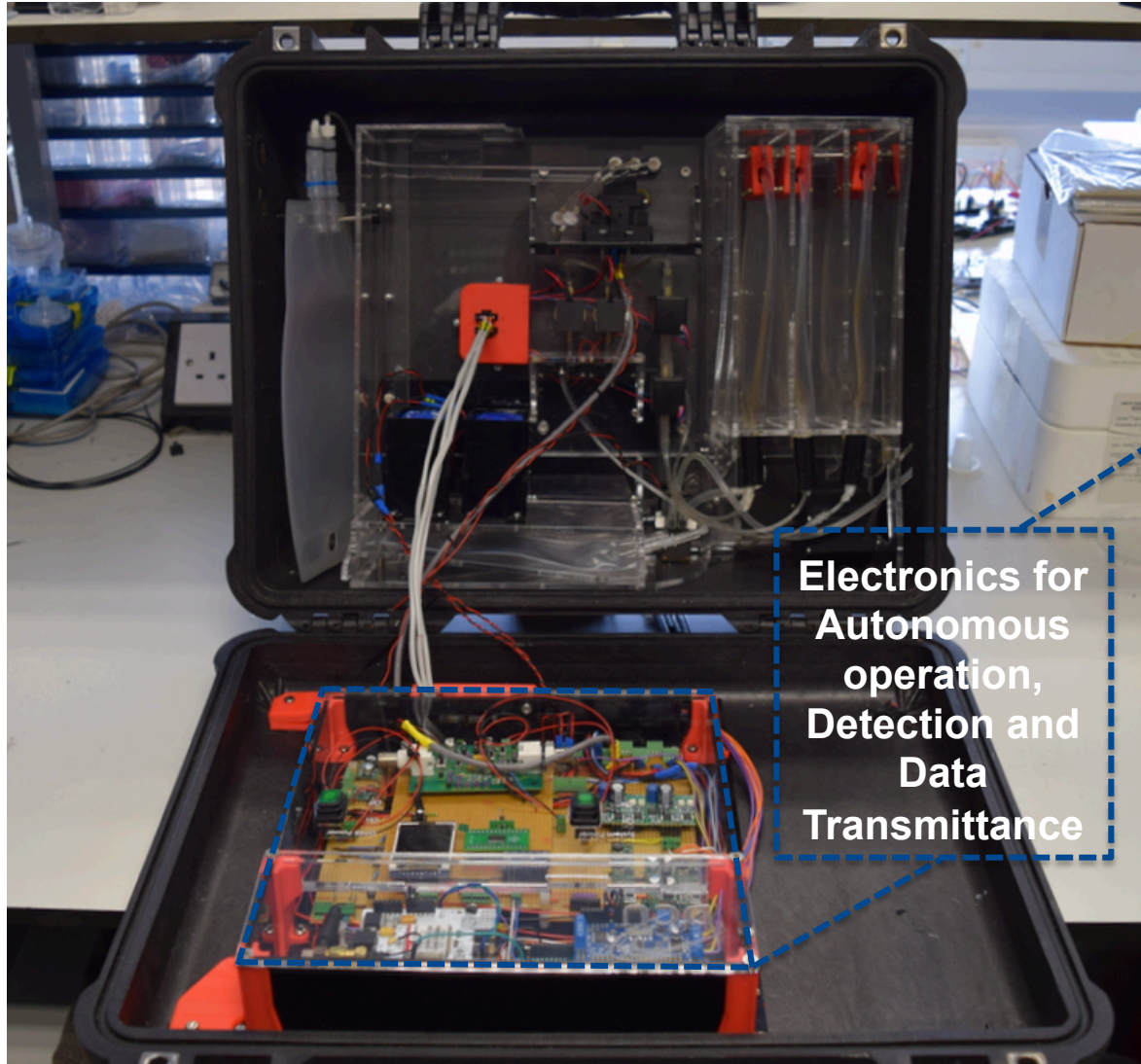
Nutrient System Schematic

8 Pumps, 12 valves, 5 Microfluidic Chips

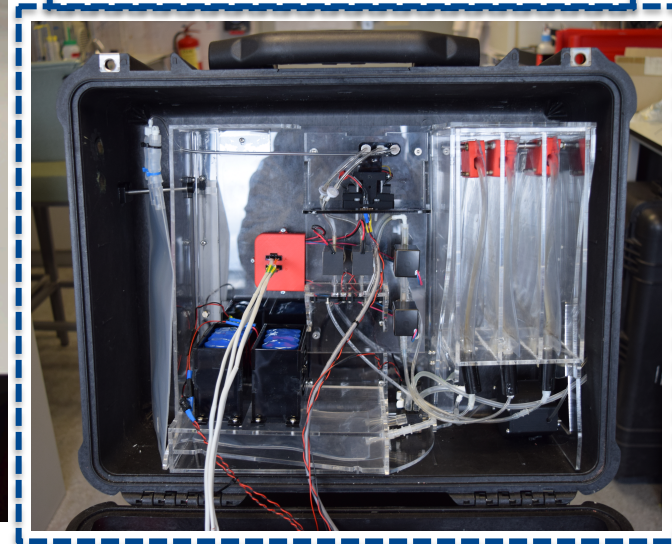
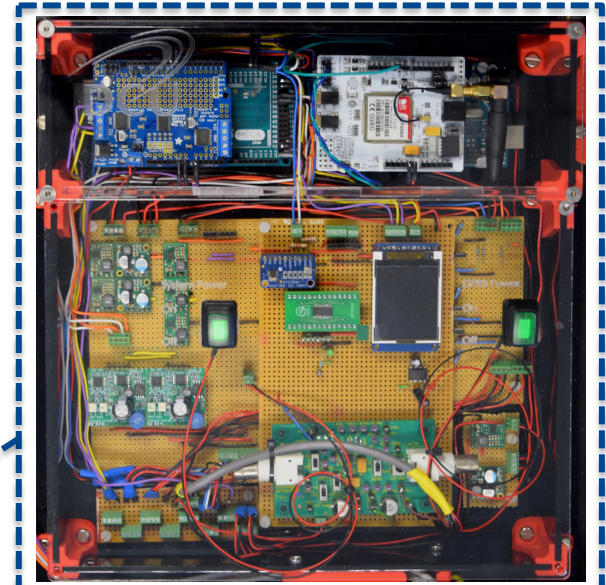




System Overview



Electronics for
Autonomous
operation,
Detection and
Data
Transmittance





Colorimetric Chemical Assay's - Phosphate



Prioritised due to

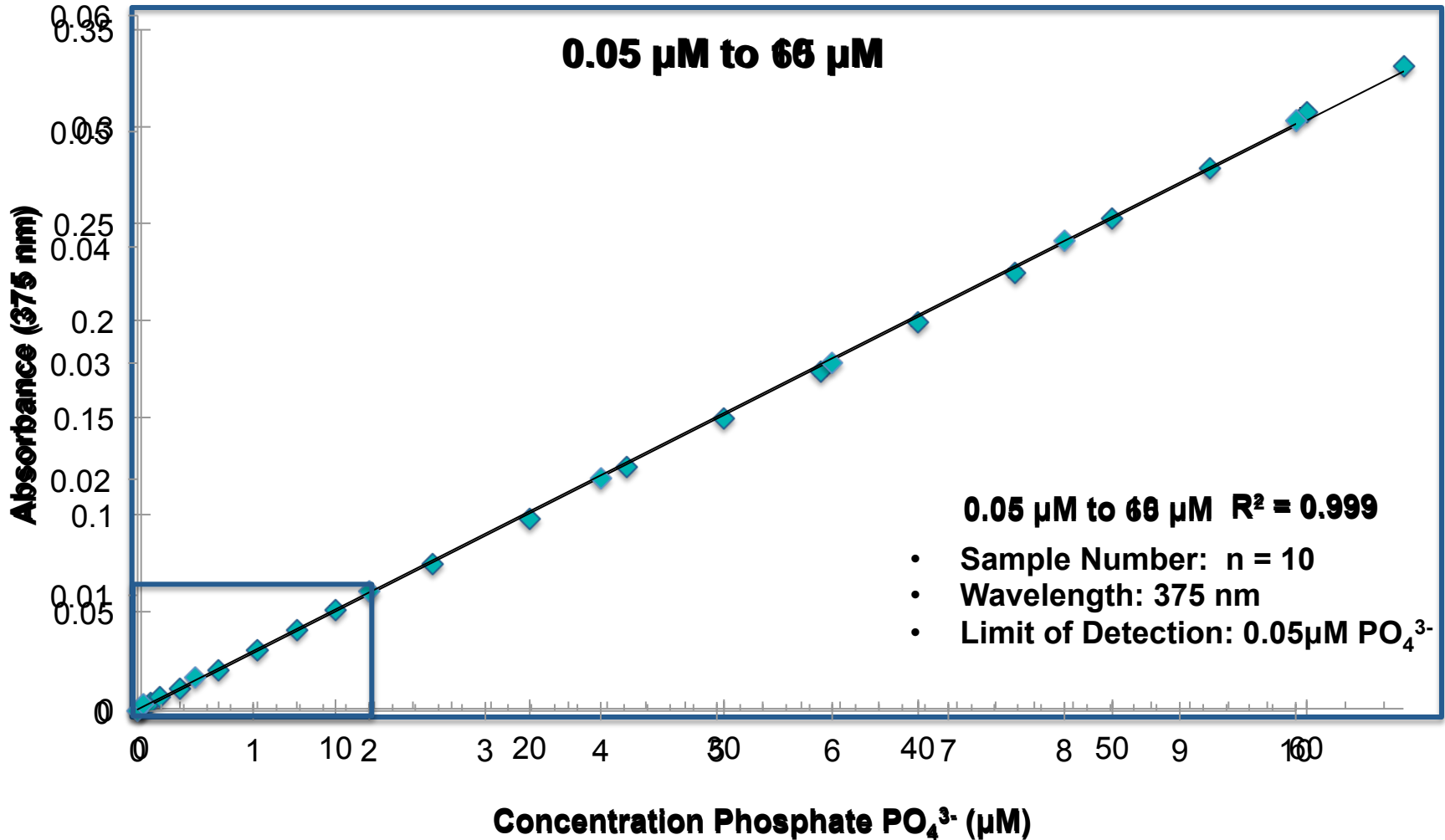
- Increasingly high demand for monitoring
- Typically the limiting nutrient in freshwater ecosystems
- Non-renewable resource – increasing attention on recovery from waste

Yellow method (vanadomolybdophosphoric acid)

- Simple colorimetric method – Single reagent 1:1 ratio
- Highly stable reagent (>1 year in solution)
- Fast reaction time
- Absorbance-based detection using UV-LED (375nm) and photodiode

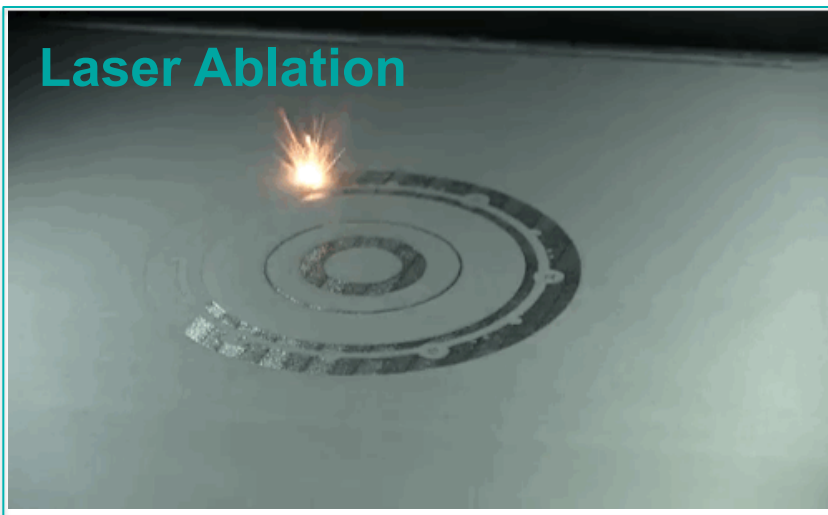
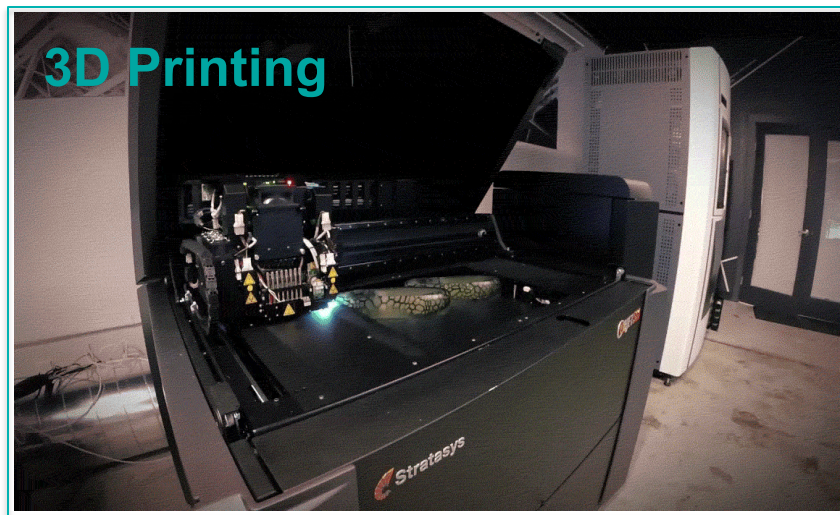
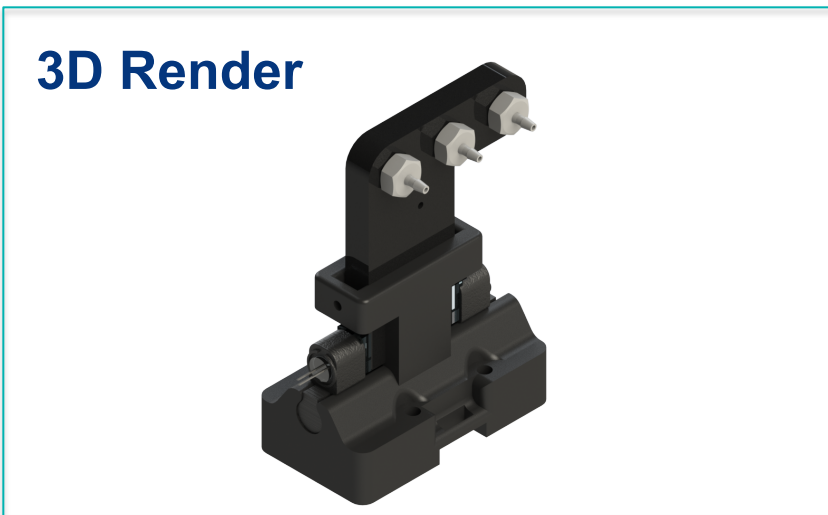
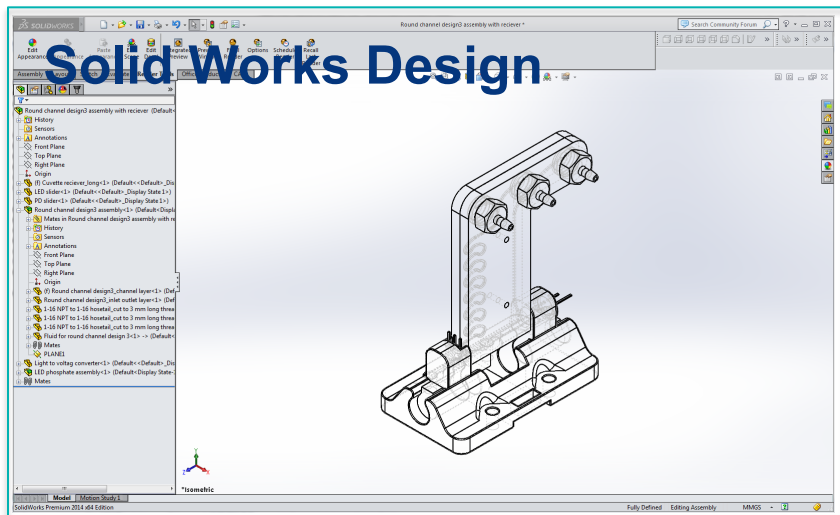


Colorimetric Chemical Assay's Phosphate Analysis in Artificial Seawater





Rapid Prototyping





Rapid Prototyped Components

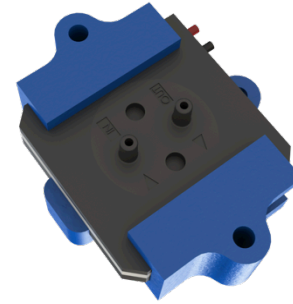


Use of 3D Printing, Laser Ablation and Micro milling techniques for rapid Prototyping

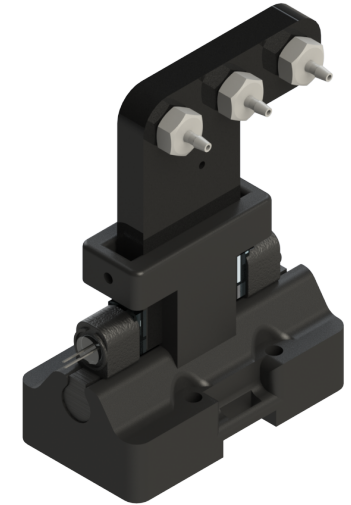
3D Render



Cuvette Holder with Led Alignment



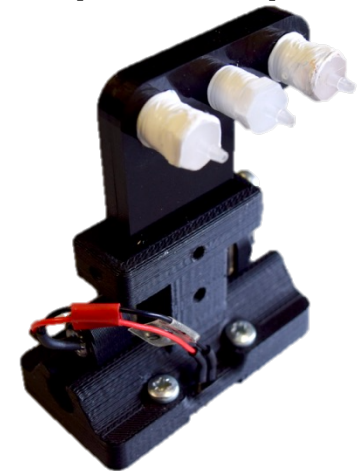
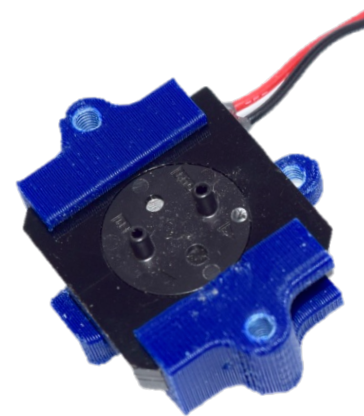
Piezo Pump Mountings



Microfluidic Optical Chip

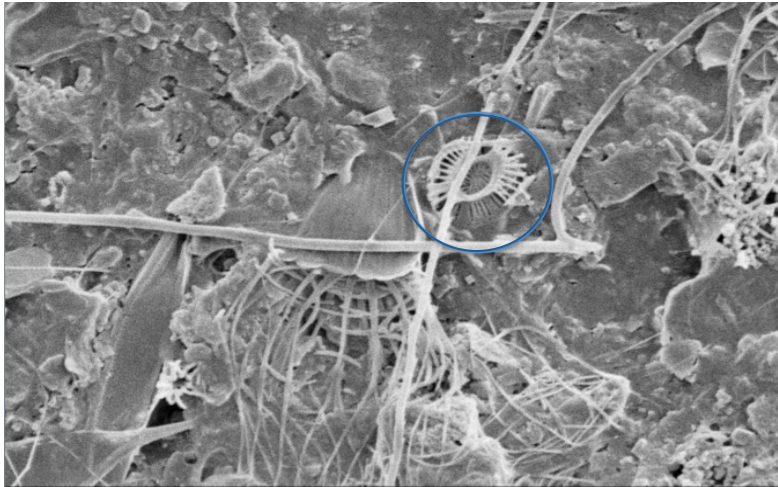
- Parts quickly and easily manufactured in house
- Reduces manufacturing time
- Reduces cost

Manufactured part



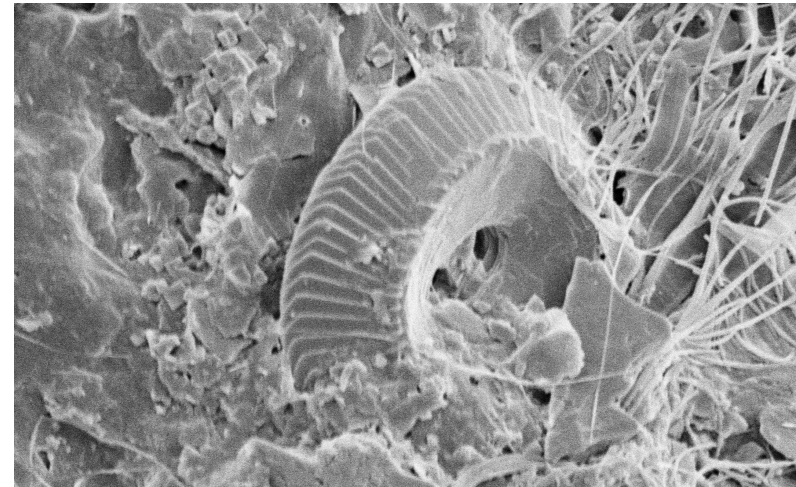


Membrane Characterisation - Diatoms



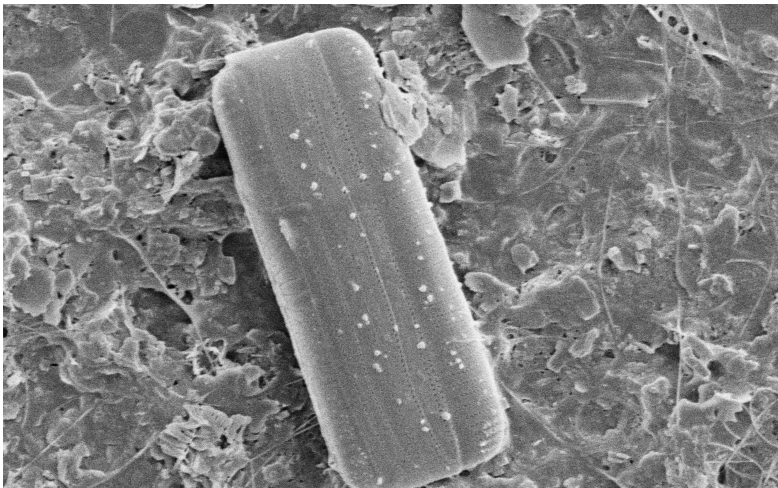
Coccolithophore - 3 Days Growth

10.0µm



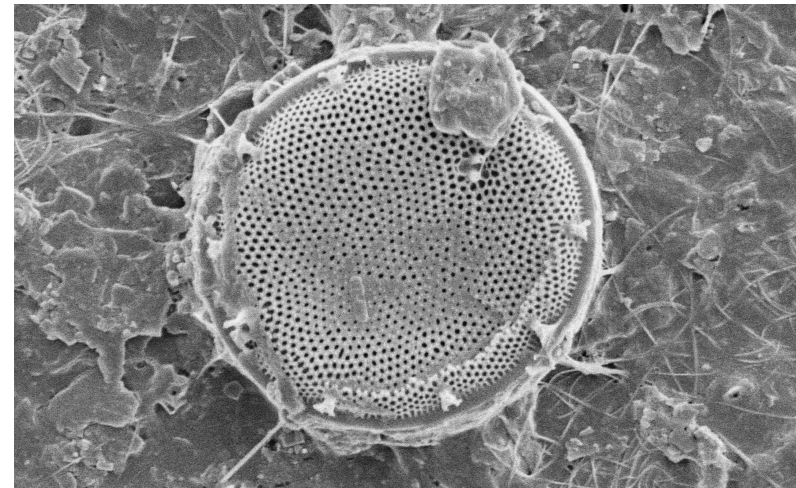
Coccolithophore - 7 Days Growth

10.0µm



Paribellus

10.0µm



Thalassiosira

10.0µm

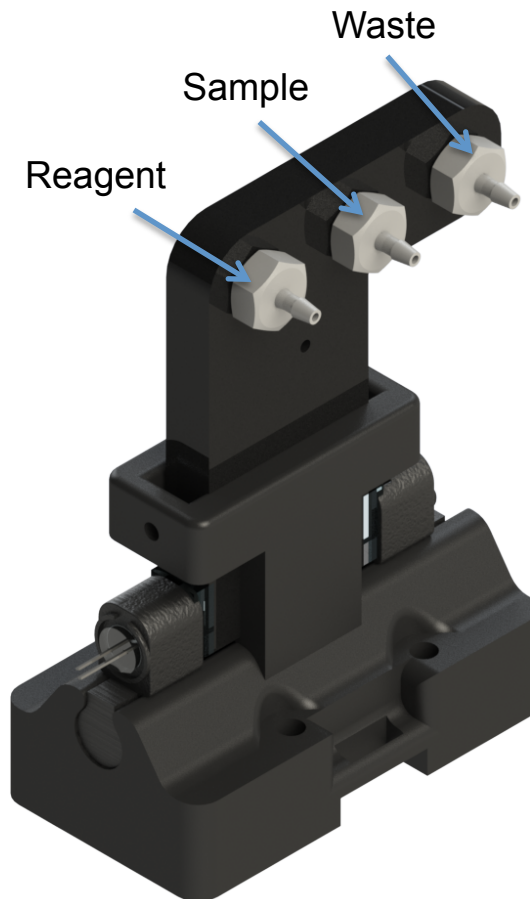


Microfluidic Detection



Overview:

- Complete integration of Fluidic Handling within a microfluidic chip.
- Manufactured using Micro milling and Laser cutting.
- Serpentine channel for Sample and Reagent Mixing.
- Minimal Fluid Volume per assay.
- Optical Detection on chip.



Detection on Chip Validated against Spectrophotometer when integrated into bench top system and Autonomous System

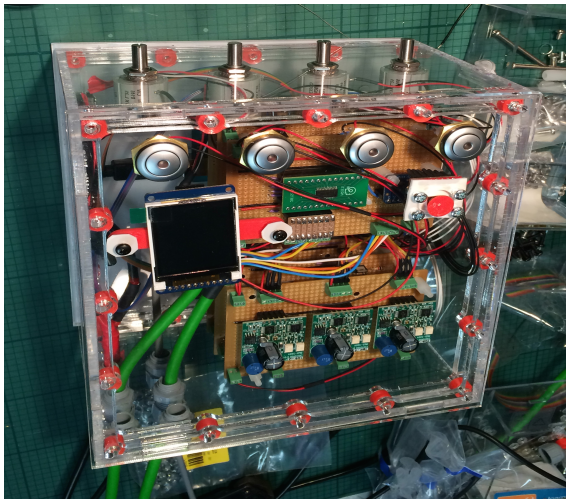


CS Nutrient Sensor



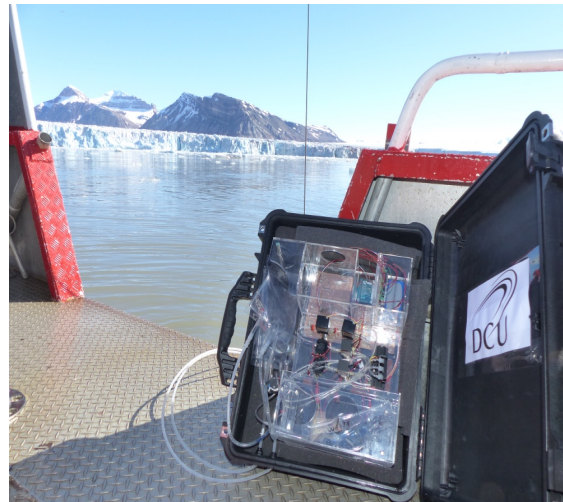
Generation 1

- Benchtop prototype
- Integrated electronics
- Optical detection
- Deployed November 2016



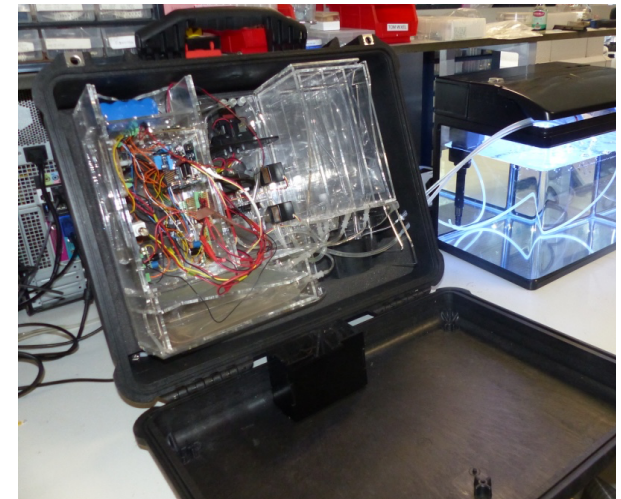
Generation 2

- Integrated fluidics
- Sample inlet
- On chip detection
- Sampling Rate every 2 hours
- Battery powered
- Deployed Ny-Alseund June 2016
- 6ml reagent per assay



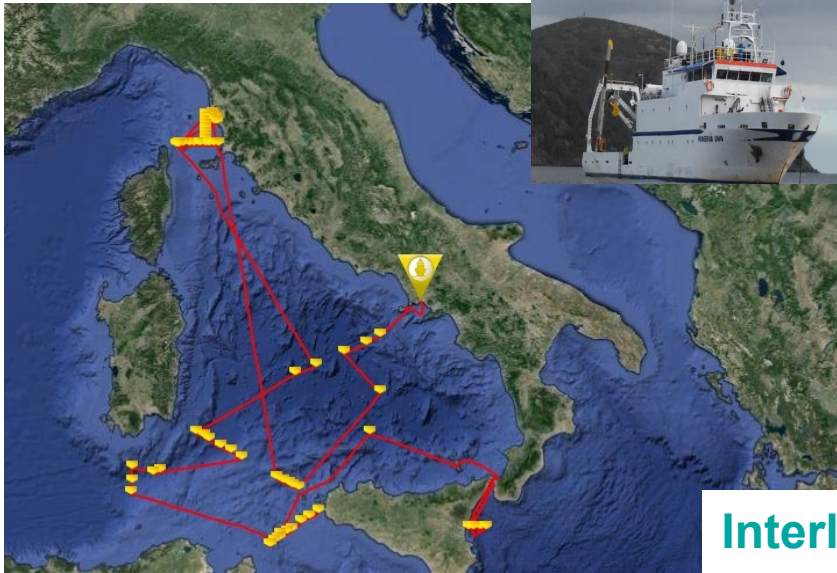
Generation 3

- Integrated fluidics
- Sample inlet
- On chip detection
- Sampling Rate every 1 hour
- Battery powered
- Wireless communication
- Deployed Milan WWTP
- 1.3ml reagent per assay

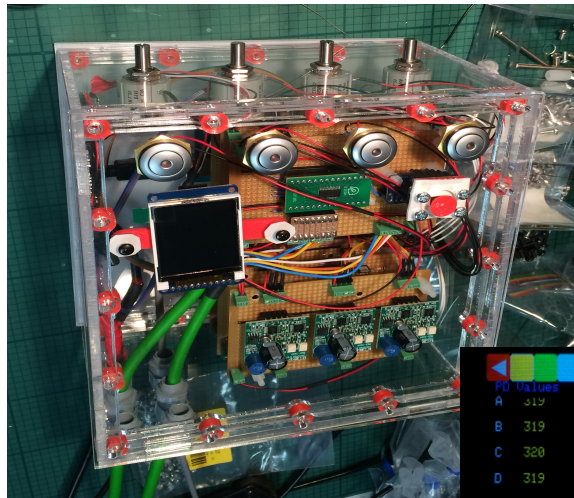




Prototype Testing - Generation 1



- Deployment onboard Ichnussa 2015 Research Cruise 24th – Dec 17th
- Water samples were collected for nutrient and heavy metal analysis, over 95 samples analysed onboard for Nitrite and Nitrate. Samples not analysed were stored at -22°C for subsequent analysis.



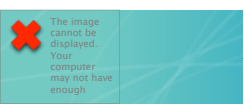
	PD Values	LED Values
A	319	12000
B	319	12000
C	320	12000
D	319	12000
E	319	12000

Interlaboratory validation

CNR-Italy All stations (48)
 DCU All stations (48)
 TelLab 10 stations



- Approximately 400 samples analysis CNR, DCU, 50 TelLabs
- Parameters: Phosphate, Nitrite , Nitrate
- DCU – CS bench-top system and UV-Vis
- Approximately 1200 measurements

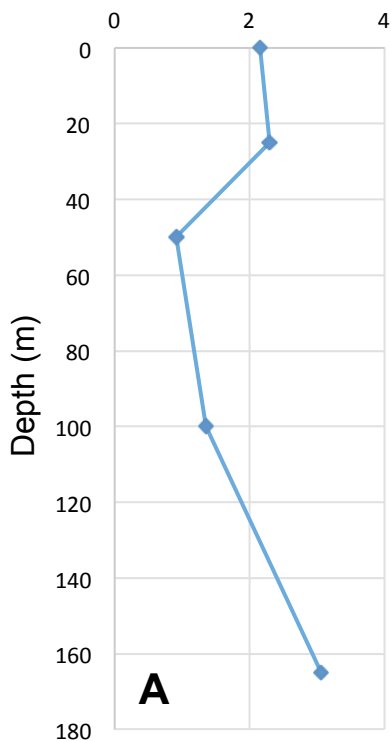




Results - Ichnussa 2015

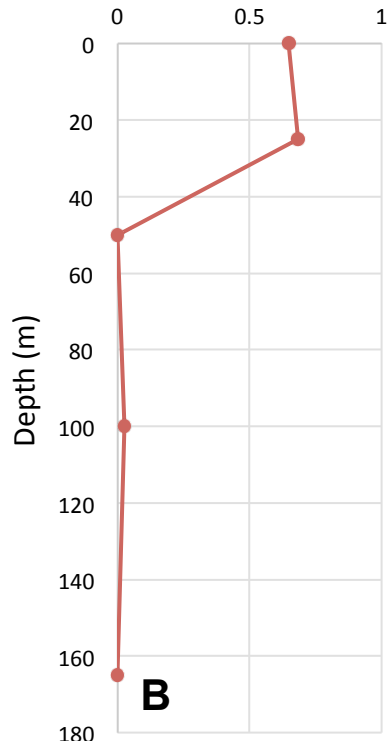
Station 432

Concentration PO_4^{3-} (μM)



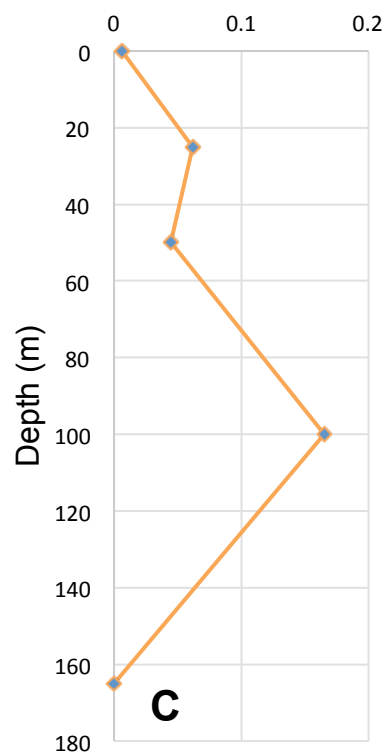
Station 432

Concentration NO_3^- (μM)



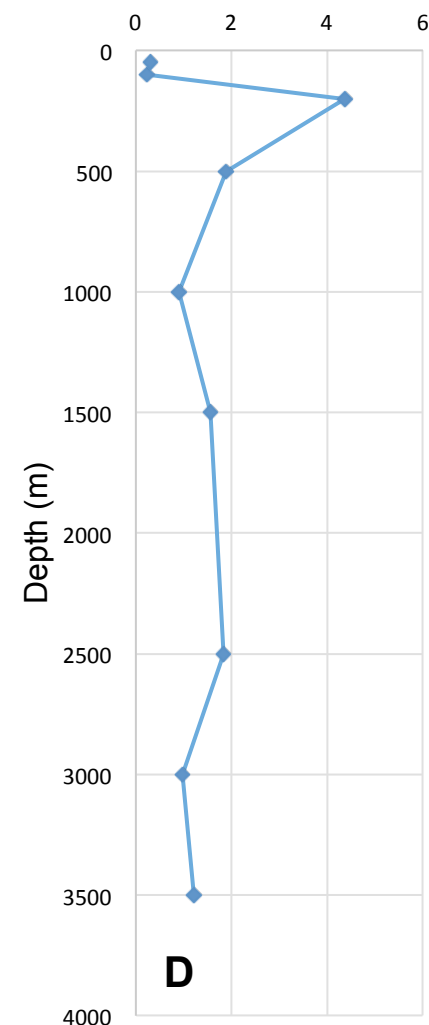
Station 432

Concentration NO_2^- (μM)



Station Geostar

Concentration PO_4^{3-} (μM)



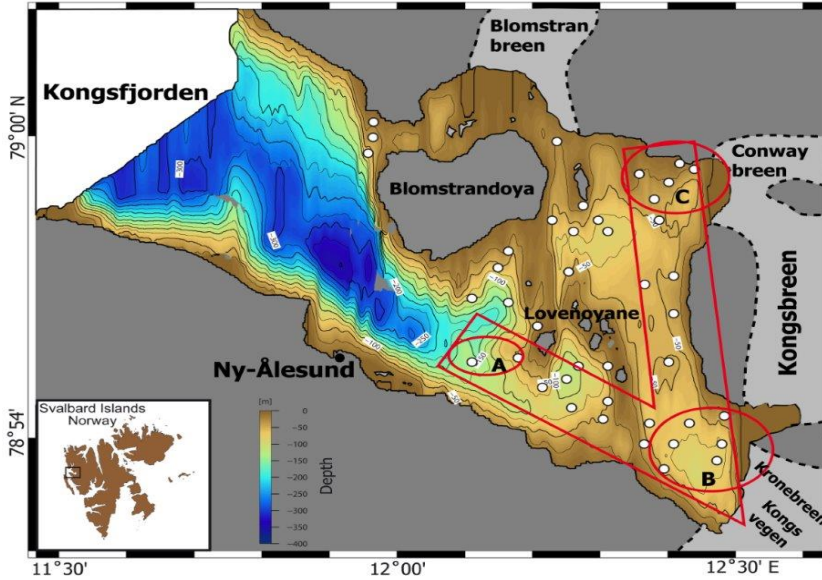
A) Phosphate **B)** Nitrate and **C)** Nitrite concentrations at along a depth profile from 0 to 166m at station 432.

D) Phosphate concentrations at along a depth profile from 0 to 3500m at station Geostar

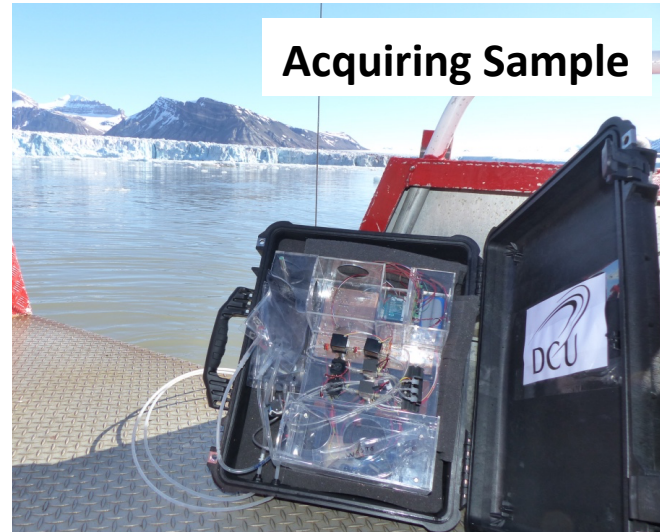


Prototype Testing – Generation 2

Deployed Ny-Alseund June 2016



Lab Validation



Acquiring Sample





Prototype Testing – Generation 2



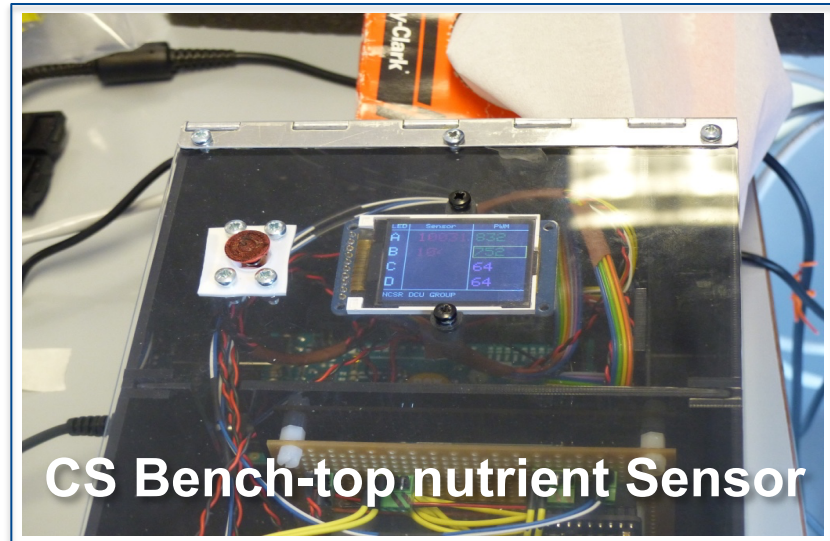
Deployed Ny-Alseund June 2016

Laboratory Testing in Ny-Alesund

- System Validation prior to deployment
- Sample was taken from 10 liters of artificial seawater spiked with a known concentration of each nutrient
- Subsamples were taken and measured using the CS bench top system



Validation at Artic Base



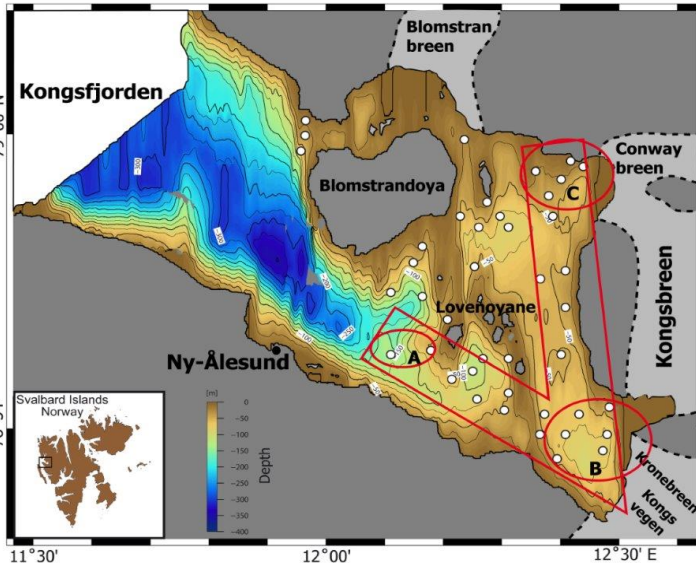
CS Bench-top nutrient Sensor



Prototype Testing – Generation 2

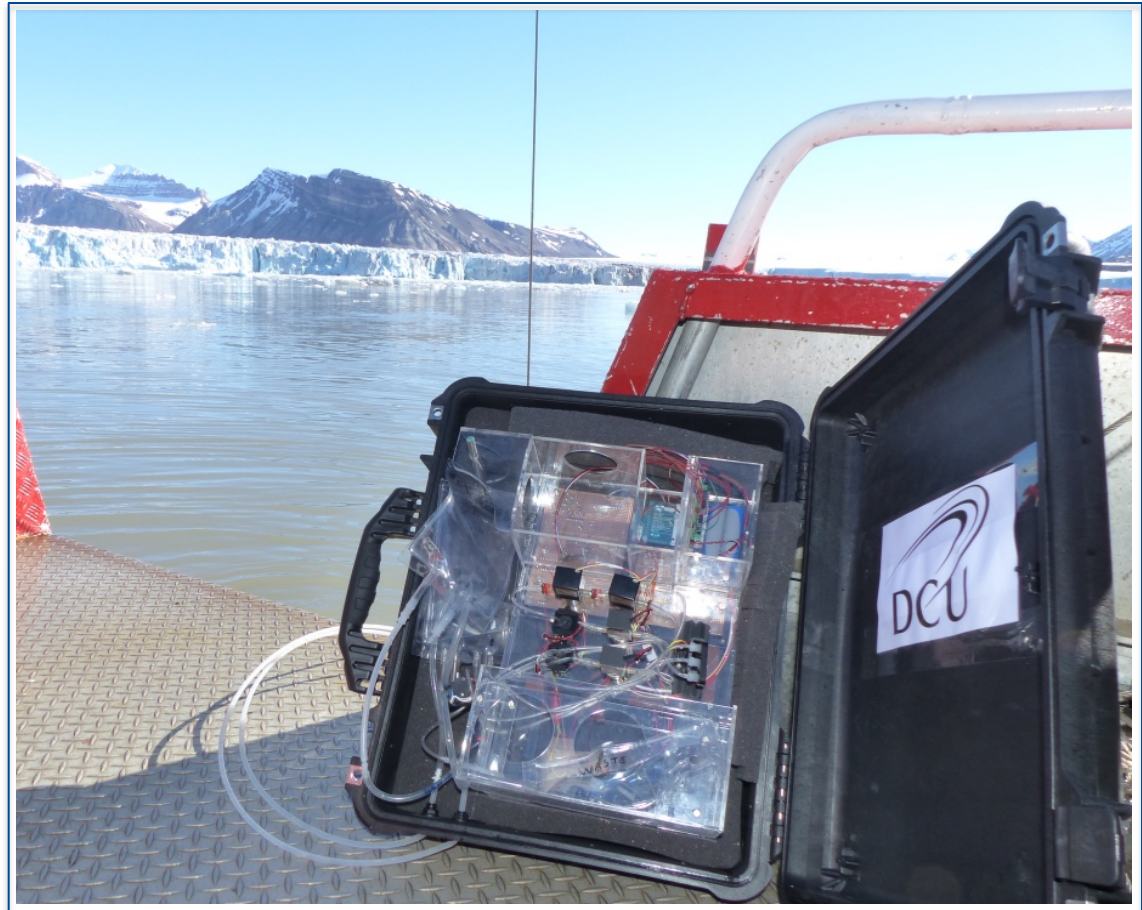


Deployed Ny-Ålesund June 2016



In-situ Measurements

- Kongsfjorden Fjord, Svalbard
- Sample Plan stations from A-B-C
- CNR Italy measuring CTD at each station



CS Deployable system acquiring samples on board the MS Teisten beneath the of Kongsvegen glacier



Prototype Testing – Generation 3



Milano San Rocco WWTP

Available Sampling Points:

1. Output water after Sand Filtration
2. Output water after the Clarifier
3. Activated Sludge (Biological Tank)
4. Input Water





Sampling Location- Sampling Point 2



Available Sampling Points:

1. Output water after Sand Filtration
2. Output water after the Clarifier
3. Activated Sludge (Biological Tank)
4. Input Water

Output water after the Clarifier: Typical Parameters

- Low detectable Nitrites
- 5mg/L Nitrates
- 1.5mg/L Phosphates
- 10mg/L Suspended Solids



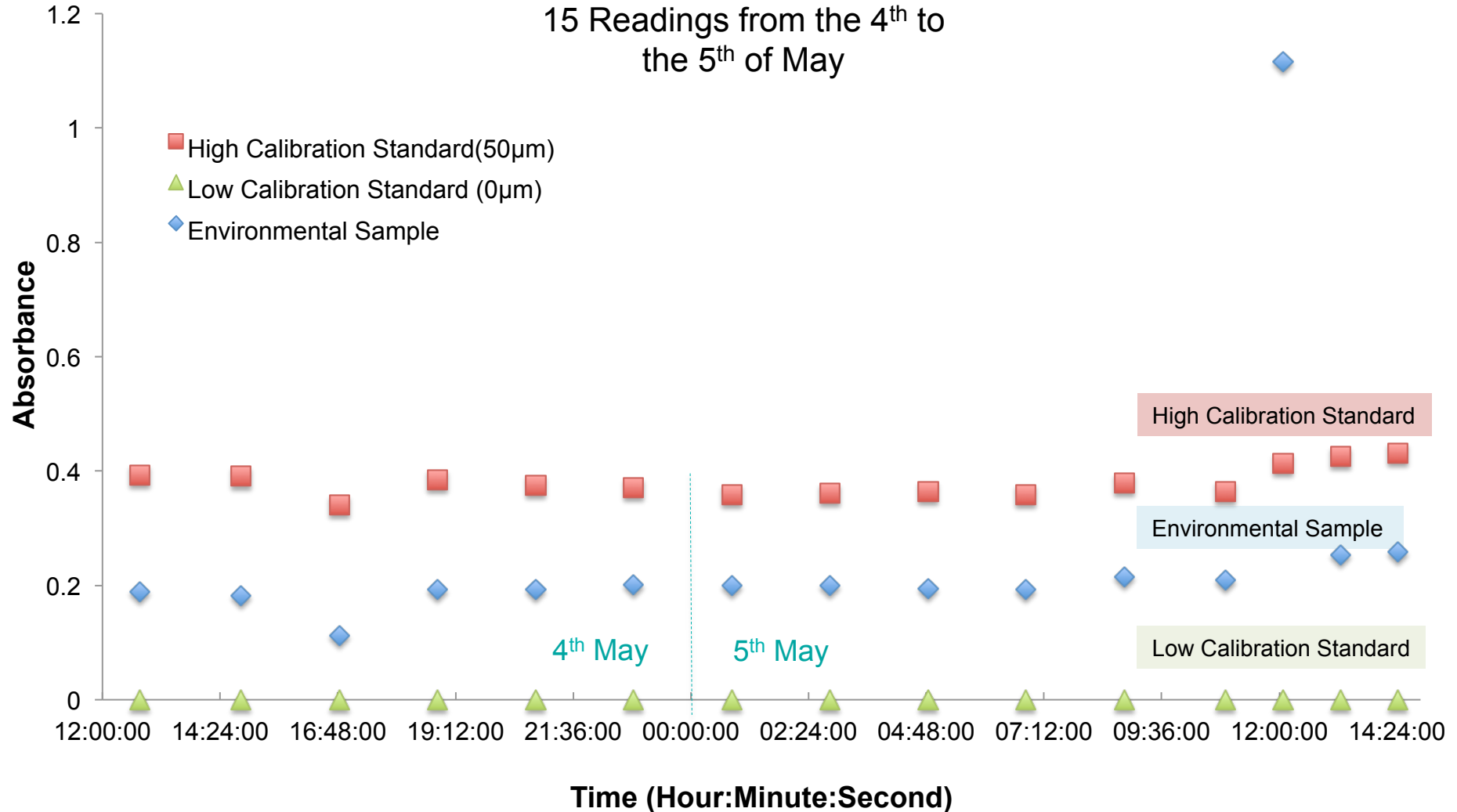


Results Obtained from Output after the Clarifier



Autonomous System Readings

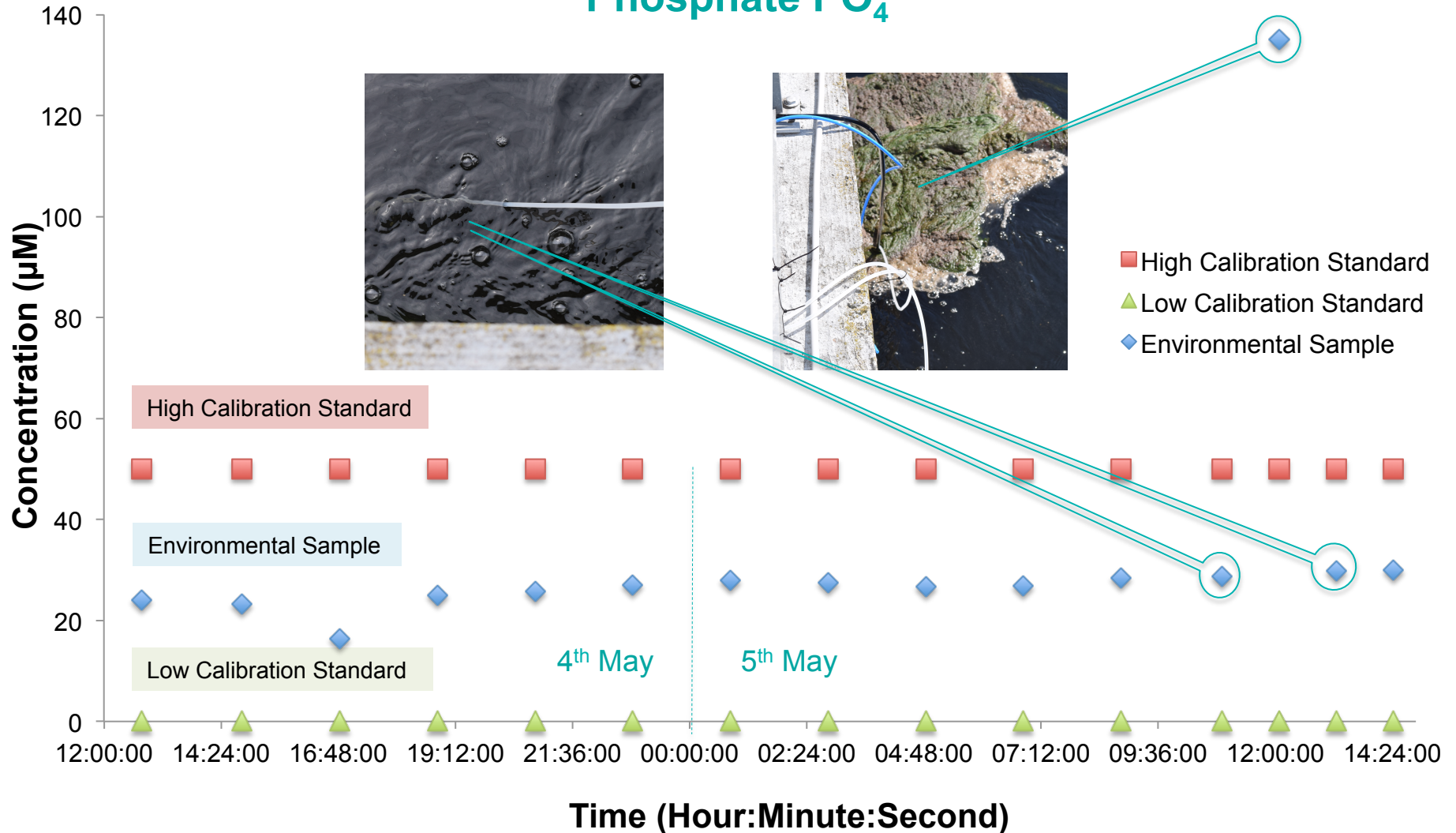
15 Readings from the 4th to the 5th of May





Results Obtained from Output after the Clarifier

Phosphate PO_4^{3-}





Inlet System- Durapore 0.45 μ M

- Approximately 5.25L sampled during deployment.
- Sample was taken for 2 ½ minutes every 2 hours.





Acknowledgements



Thanks to:
Prof. Dermot Diamond, Dr. Margaret McCaul, Adaptive Sensors Group, All in N205 DCU

This Research is co-funded by the European Regional Development Fund (ERDF) under Ireland's European Structural and Investment Funds Programmes 2014-2020

