



# Microfluidic platform for multi-parameter water quality analysis

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# Outline

- **Introduction**
- **System design and development**
- **Phosphate sensor design and deployments**
- **Other parameters**
- **Future plans & Conclusions**

# Introduction

## Motivation

- Monitoring of environmental water quality is predominantly based on manual sampling followed by laboratory analysis
- Expensive → Limited in terms of frequency and number of locations
- Not sufficient to satisfy the demands of environmental legislation (e.g. EU WFD)



## Autonomous sensors

- Can provide high-frequency data from a larger number of locations with lower cost

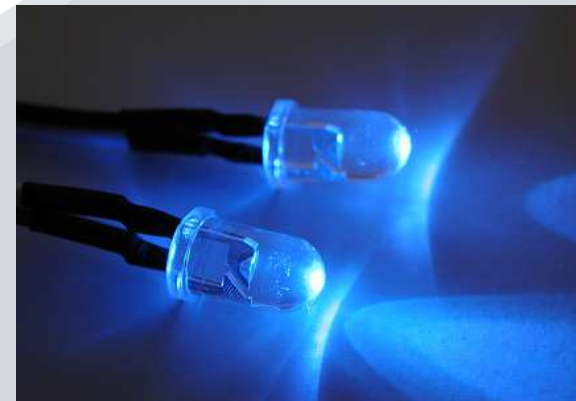
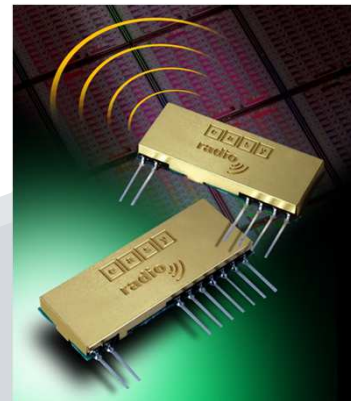
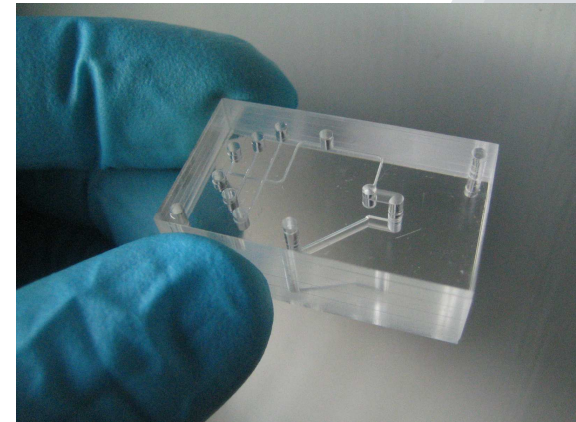
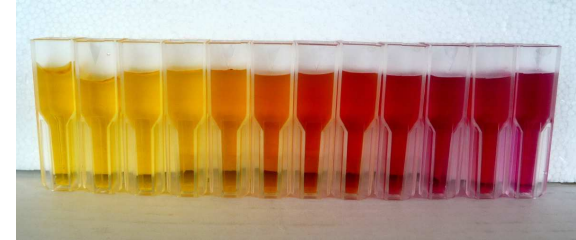


# System requirements

- Reliable and sensitive detection system
- Long deployable lifetime ↔ Low power consumption
- Wireless communication of data to user
- Rugged and portable design
- Low cost, Low maintenance

# Approach

- Colorimetric reagent chemistry
- Microfluidic technology
- LED-based detection systems
- Communication by GSM / short range radio (e.g. ZigBee)



# Key factors in system design

## Chemistry

- Sensitive detection (mg/L -  $\mu$ g/L)
- Reagent stability
- Simple methods (number of reagents/steps)  
→ simple, robust microfluidic chips

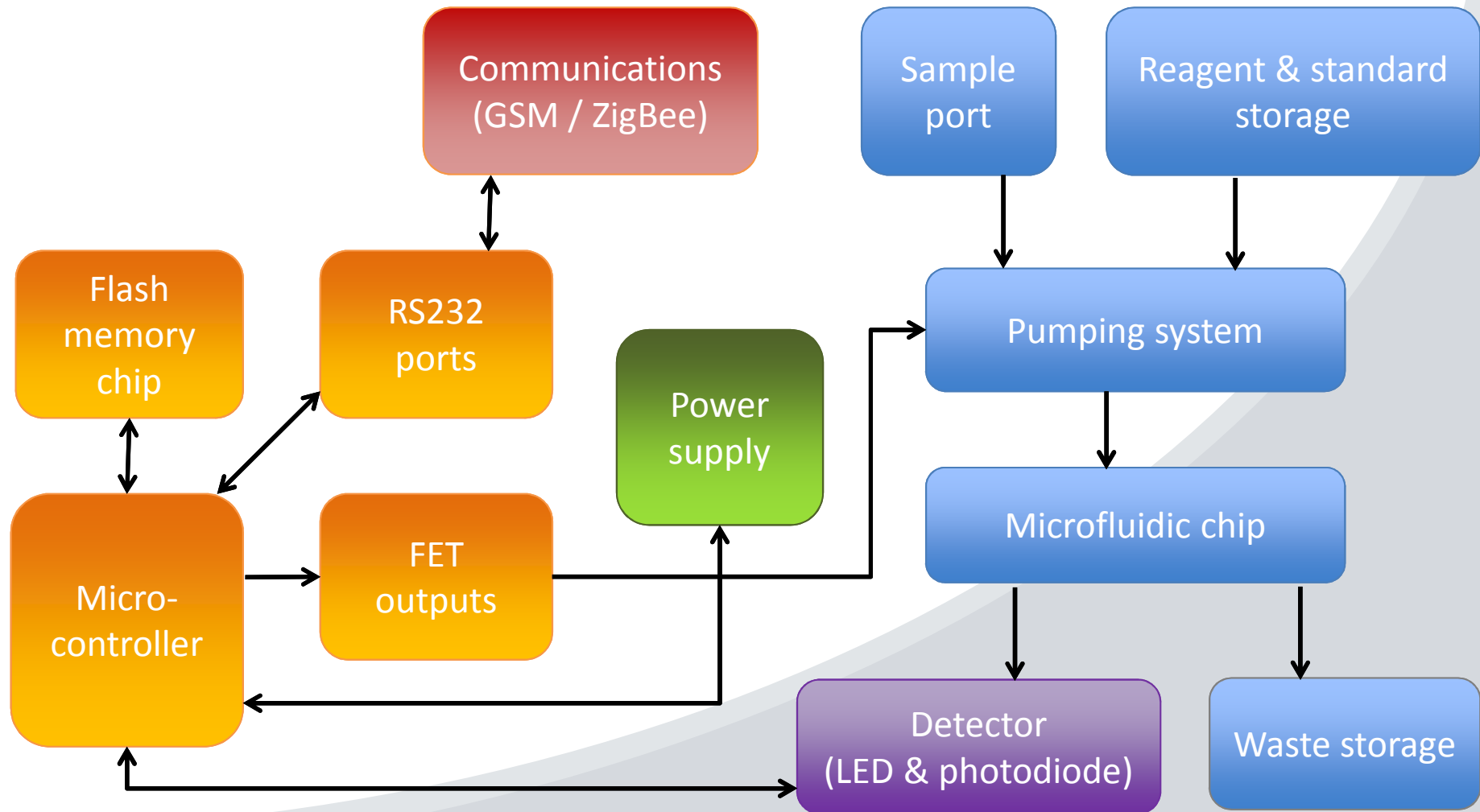
## Detectors

- LED & photodiode or Paired Emitter Detector Diodes (PEDDs)
- Sensitive, Low power consumption, Low cost

## Communications

- Application dependent (range, frequency, power demands)

# System design



# Phosphate analyser

## 3 stages of development

- Demonstration of method (chemistry & detector) in microfluidic manifold
- Development and field testing of prototype system
- Commercialisation (Episensor Ltd., Ireland)

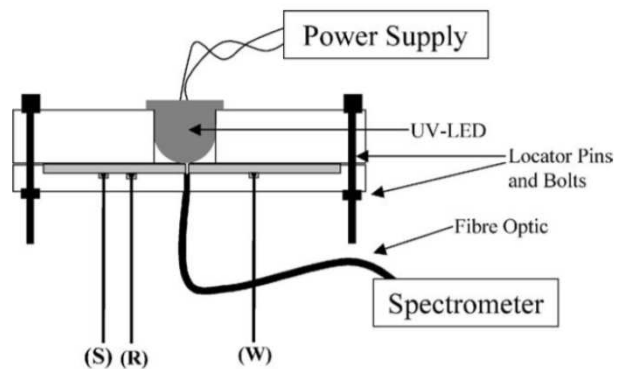
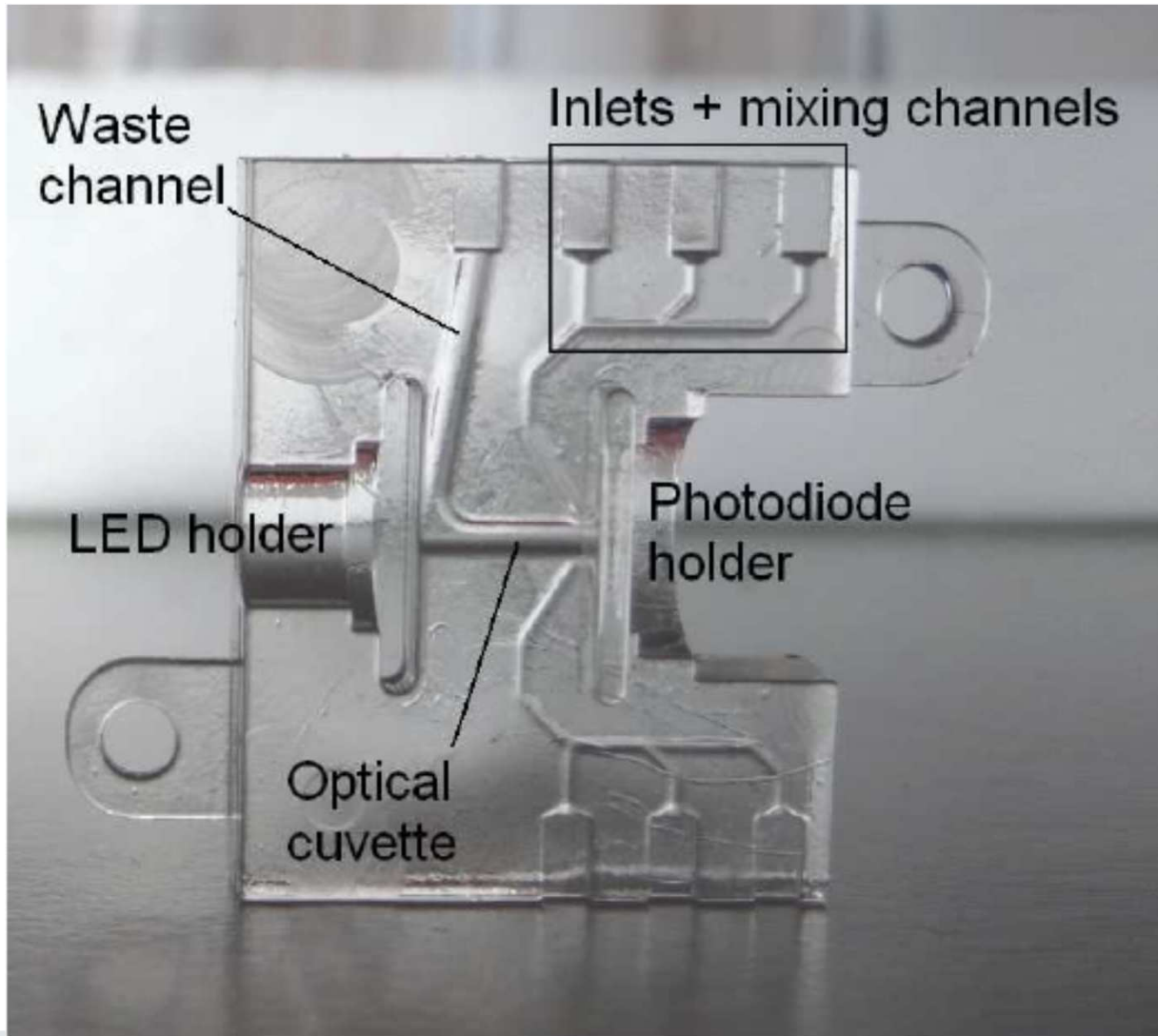


Fig. 1. Cross-section schematic of the optical alignment through the microfluidic chip.



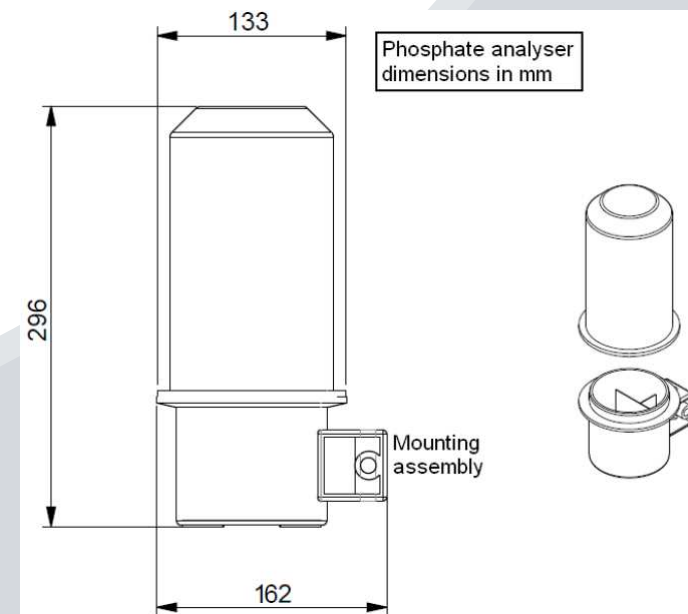
# Phosphate sensor



- (1) Sample inlet;
- (2) Control board and detection system;
- (3) Dual channel peristaltic pumps;
- (4) Reagent bags;
- (5) IP68 enclosure

# Specifications

- Minimum sample interval: 15 minutes
- Linear response range: 0-20 mg/L PO<sub>4</sub><sup>3-</sup>
- Limit of detection: 0.20 mg/L PO<sub>4</sub><sup>3-</sup>
- Up to 1000 assays before reagent/battery replacement
- Weight 1.8 kg with full reagent load

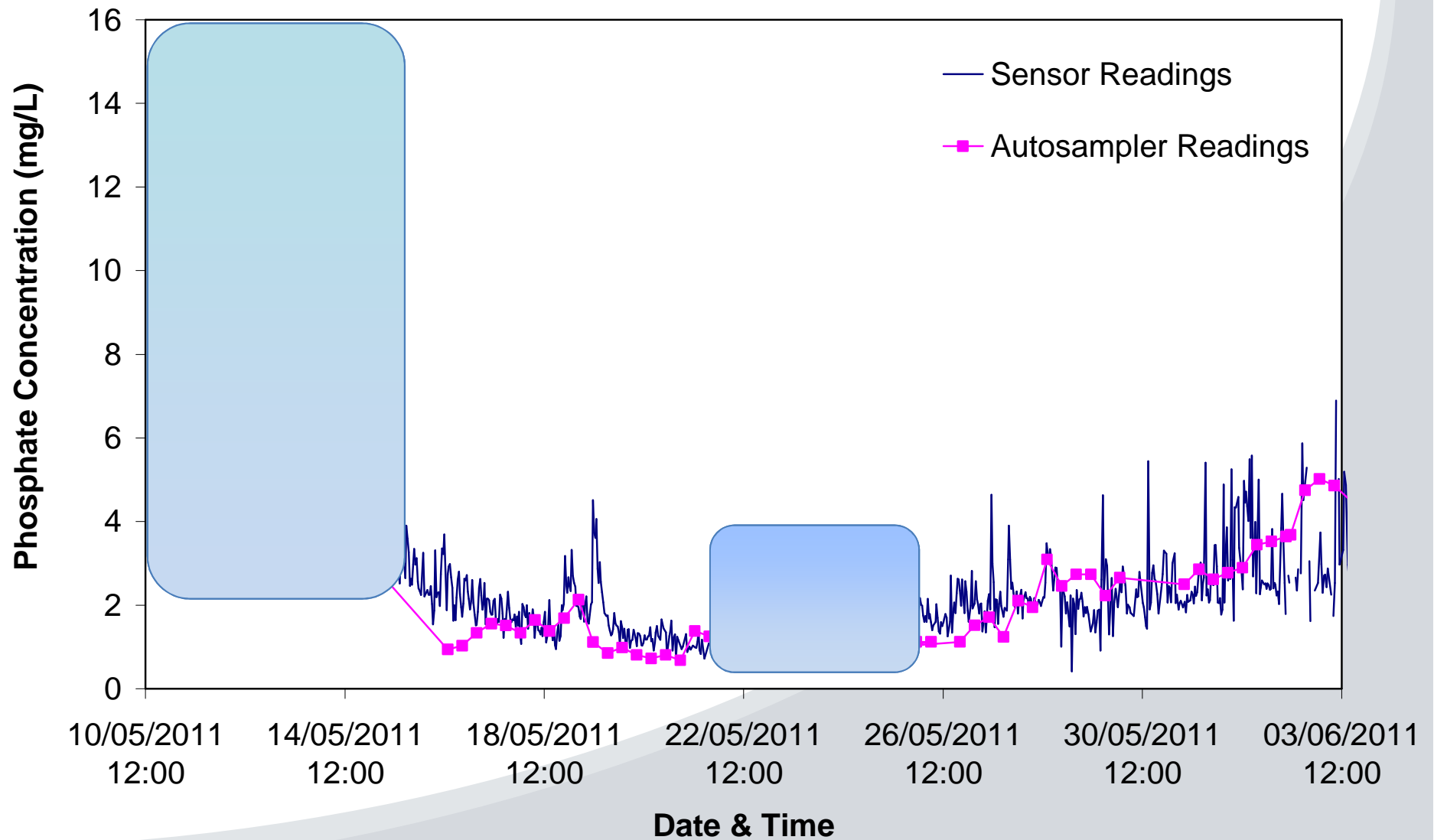


# Deployments (i) Wastewater



- WWTP in Co. Kildare, Ireland
- Sensor installed in effluent discharge tank
- 30 min sample interval
- Autosampler collecting 24 samples/week for validation

# Deployments (i) Wastewater

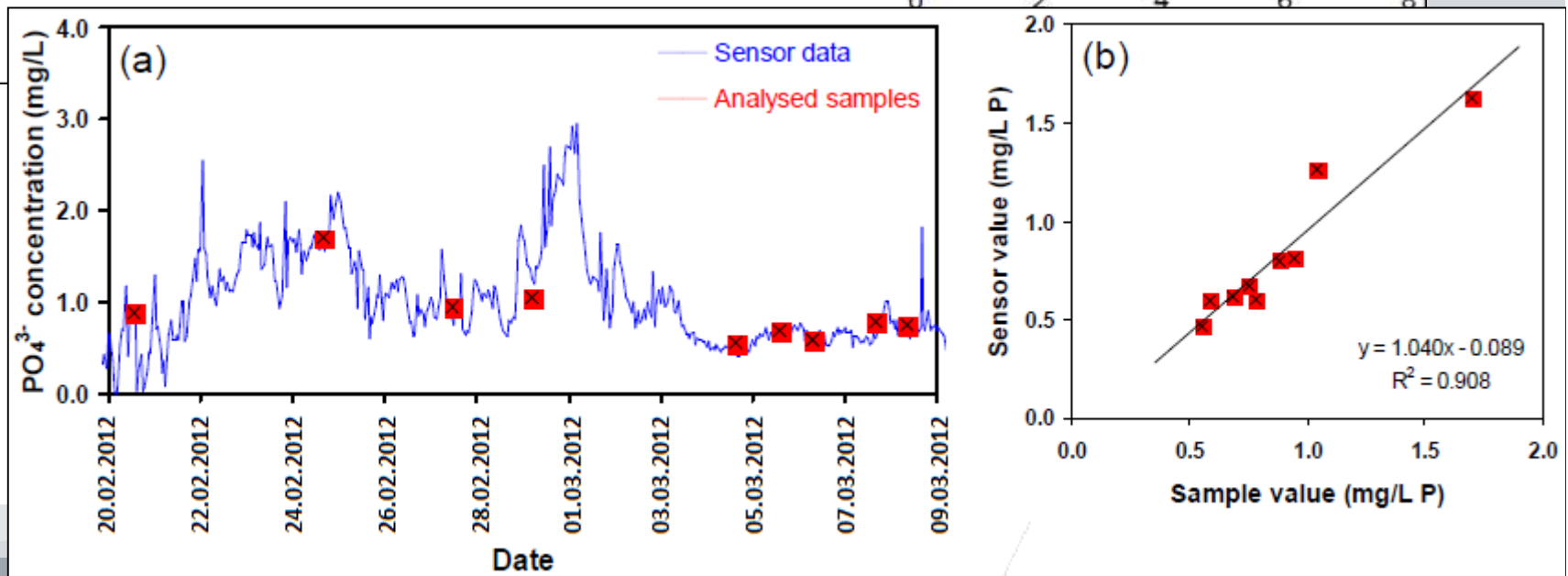
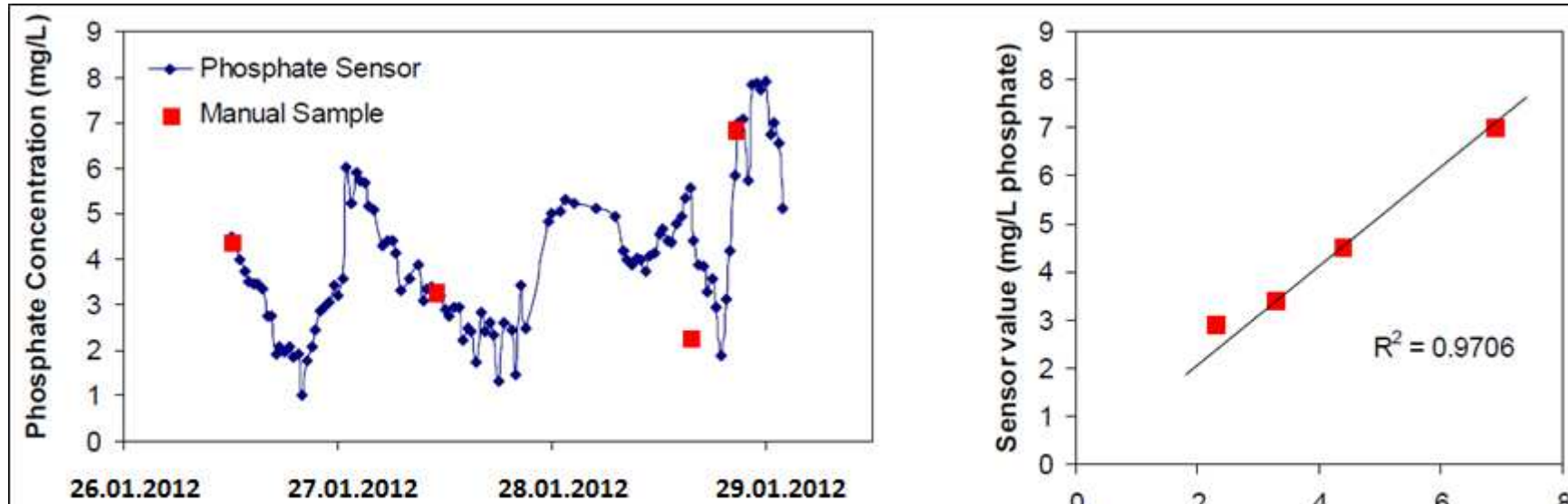


# Deployments (ii) Estuary



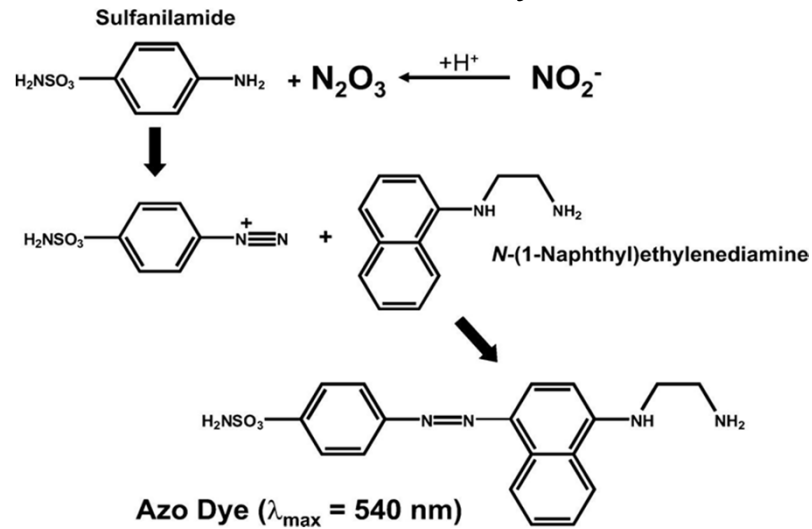
- Broadmeadow Water
- Enclosed estuary north of Dublin City
- Highly impacted - municipal wastewater + agricultural inputs

# Deployments (ii) Estuary

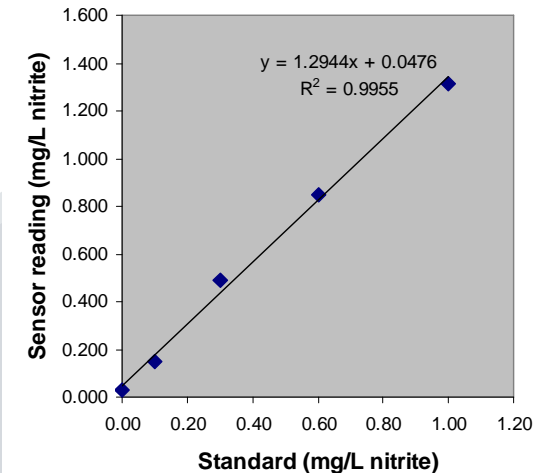


# Nitrite detection

## Griess assay



- Single reagent  $\rightarrow$  can be incorporated into platform with minimal changes
- Detection at 540 nm
- Detection limit 5  $\mu\text{g/L}$

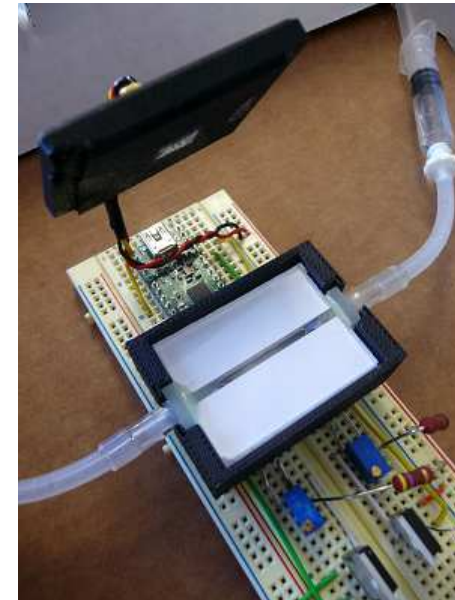
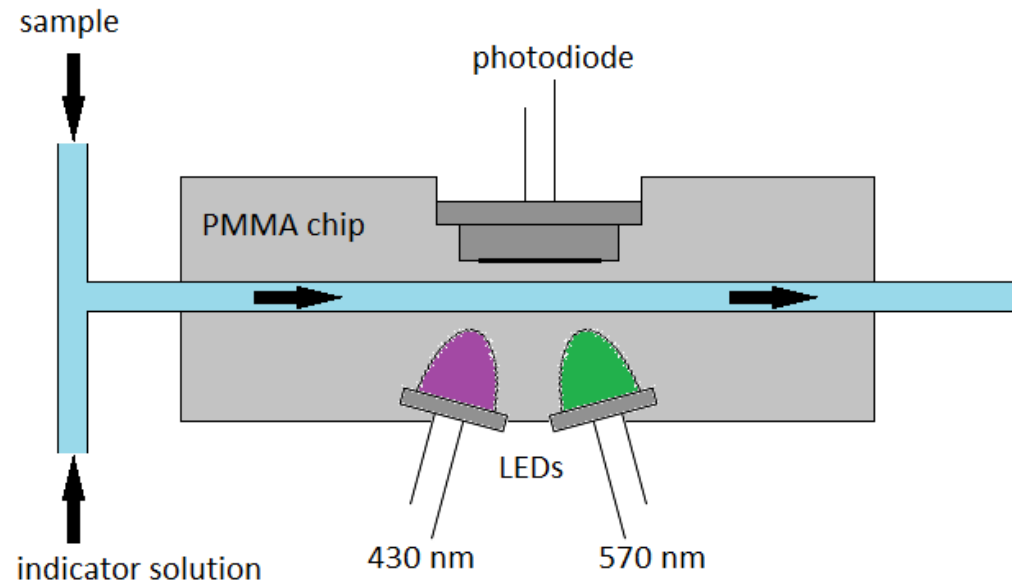


# Nitrite detection – blind test

Sample Reference/Type	Sample ID	Sample Concentration 17/02/2012 by IC	Sample Concentration 21/02/2012 by HACH (LL)	Sample Concentration 21/02/2012 by IC	<u>DCU results</u> <u>22/02/12</u>
100413	A	<0.2	0.016	<0.2	0.115
100413 Spiked with 1mg/l NO <sub>2</sub>	A*	0.98	1.020	0.92	1.190
100343	B	<0.2	0.008	<0.2	0.023
100343 Spiked with 1mg/l NO <sub>2</sub>	B*	0.94	1.006	0.85	0.914
100291	C	<0.2	0.012	<0.2	0.161
100291 Spiked with 1mg/l NO <sub>2</sub>	C*	0.96	1.002	0.92	1.342
1mg/l NO <sub>2</sub> standard	D	0.89	0.870	0.77	1.142
2mg/l NO <sub>2</sub> standard	D*	1.71	1.865	1.72	1.920

Samples and external analyses supplied by TE Laboratories Ltd.

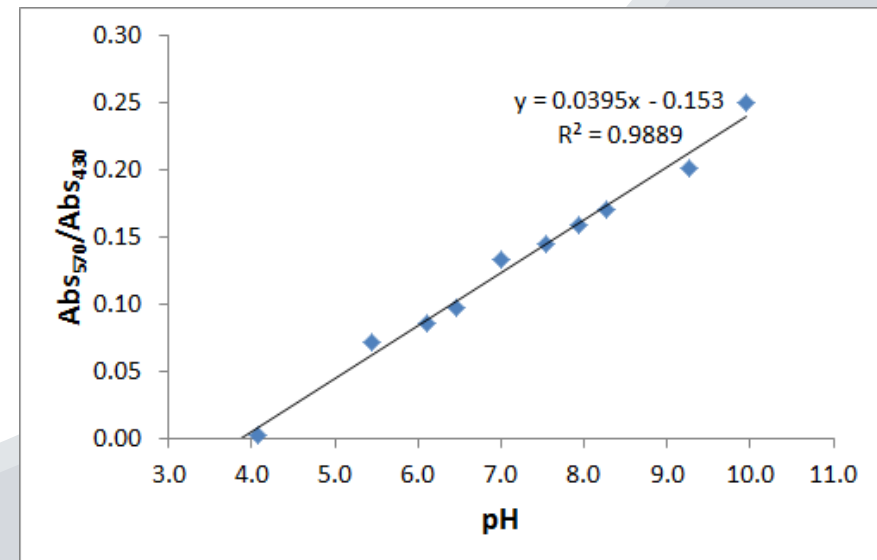
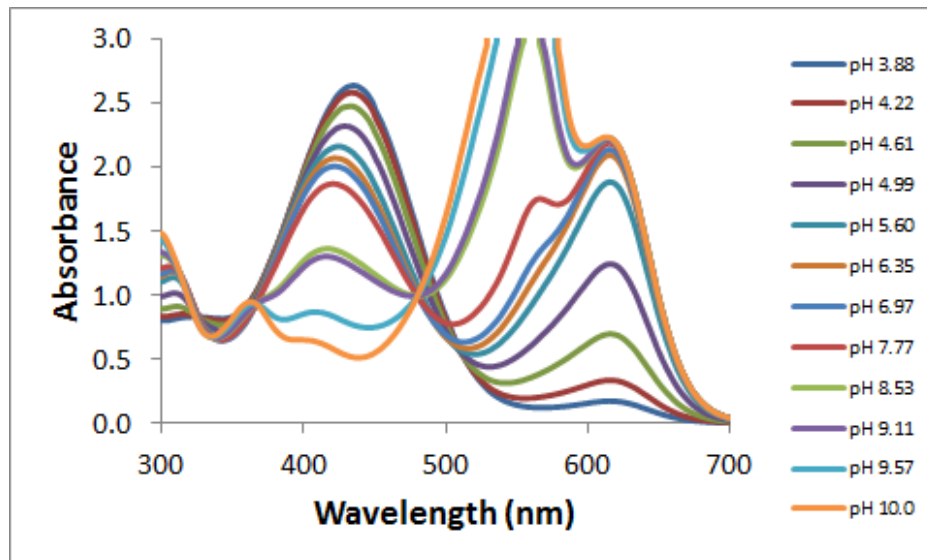
# pH measurement



- Mixed indicator solution
- Dual LEDs with adjustable intensity
- Photodiode detector

# Mixed indicator solution

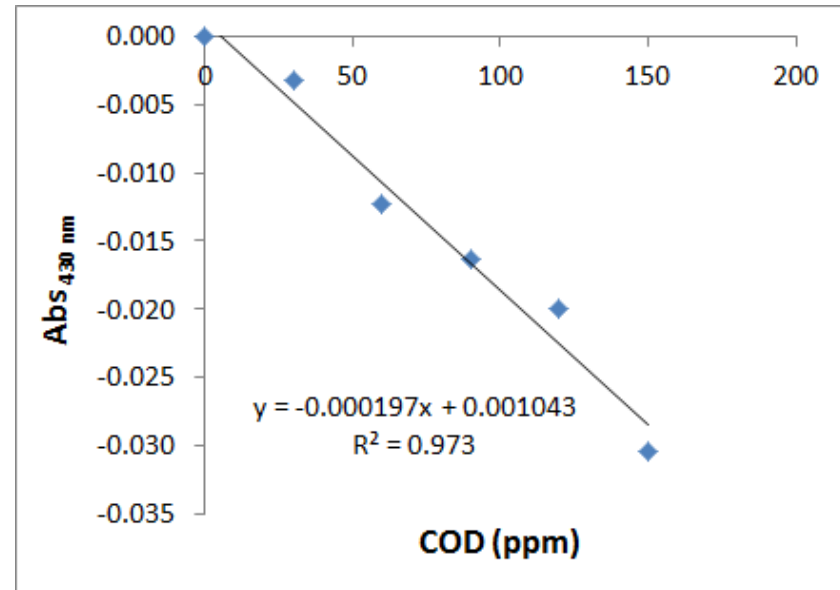
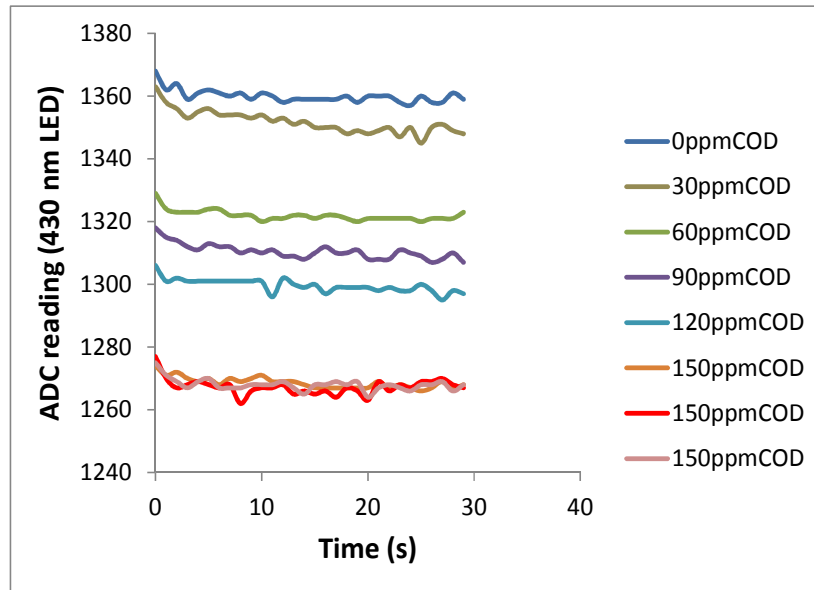
- Colour change over range pH 4 - 10
- 3 options for detection wavelength
- Linear response using ratio of 430 and 570 nm peaks



# Chemical Oxygen Demand (COD)

- Standard COD method based on oxidation using potassium dichromate, conc.  $\text{H}_2\text{SO}_4$ , heating to  $150^\circ\text{C}$  for 2 hours
- Colour change from chromate ion ( $\text{Cr}_2\text{O}_7^{2-}$ , orange) to chromic ion ( $\text{Cr}^{3+}$ , green)
- Monitor decrease in chromate peak at 430 nm

# Detector output

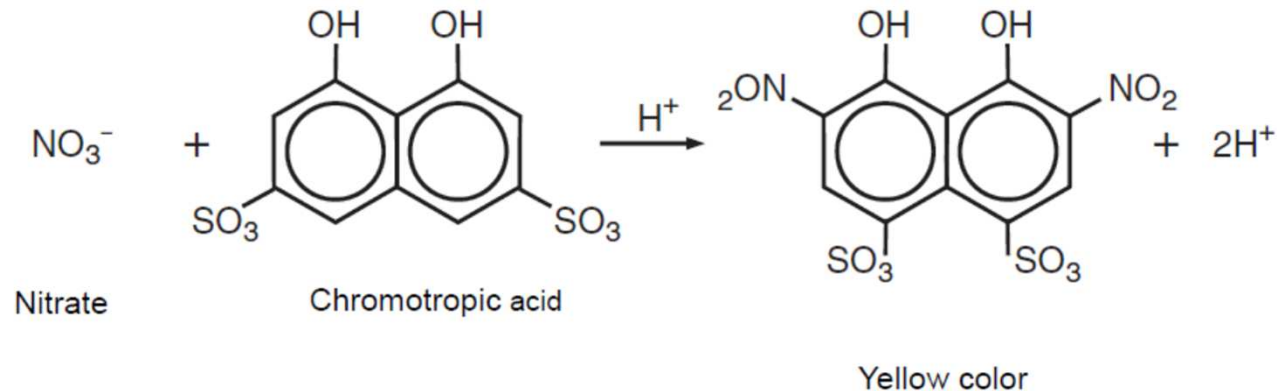


## Issues

- Strong acid → resistant materials → more expensive
- Heating → high power consumption

# Nitrate detection

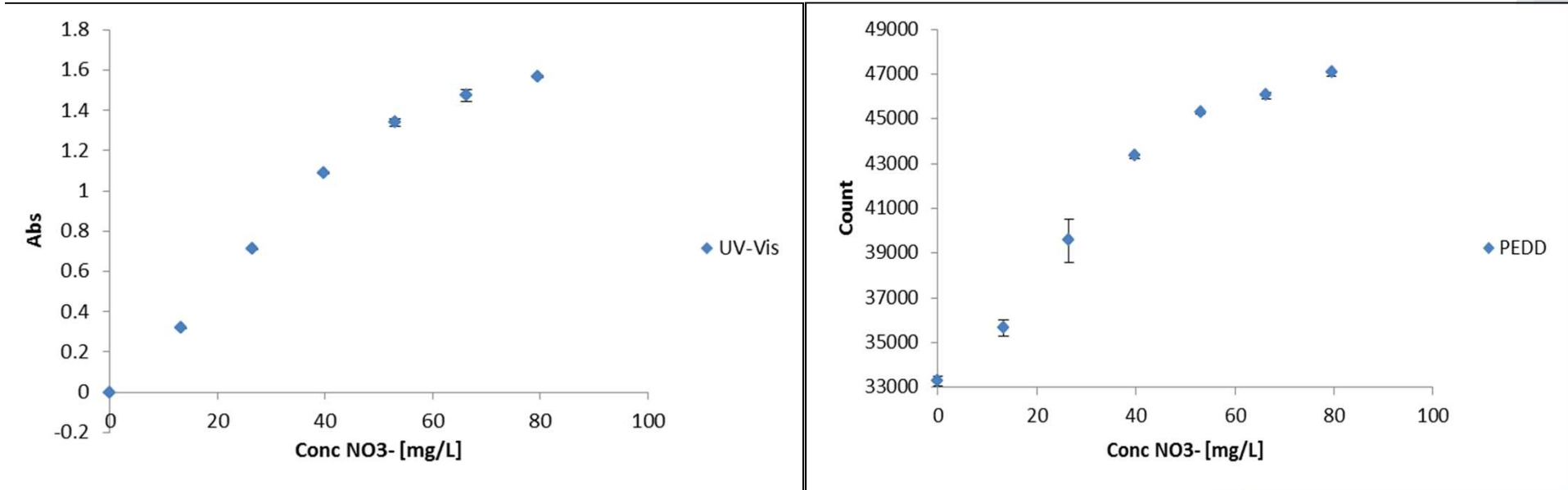
## Chromotropic acid method



- Simplified version of literature method\* in which reagents (CA + conc.  $\text{H}_2\text{SO}_4$ ) added separately with cooling
- Reagents can be combined into single solution, cooling step eliminated

\* Ryan, J., Estefan, G. and Rashid, A. 2001. *Soil and Plant Analysis Laboratory Manual*. Second Edition. Syria. ICARDA and NARC 2001.

# Calibration

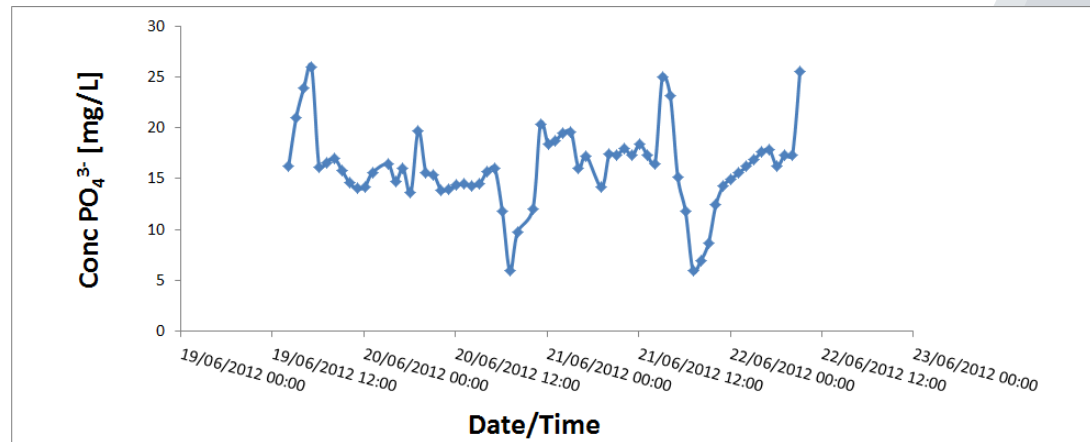


- Calibration curves by UV-vis and LED-based detection
- Linear response to approx . 50 mg/L
- Suitable for environmental and drinking water applications

# Future work

## Phosphate

- Long term deployment of multiple sensors at sites of interest
- Recently installed at constructed wetlands treatment system



## pH, Nitrite

- Field-testing of prototype units



# Future work

## COD

- Optimisation of detection method in microfluidic system using dual wavelength detection (430 and 600 nm)
- Development of prototype for wastewater monitoring

## Nitrate

- Optimisation of chromotropic acid method
- Development of prototype (drinking water)

# Conclusions

Deployable sensor for phosphate has been developed and successfully field tested in wastewater, freshwater and estuarine conditions

Prototypes for nitrite and pH have been developed and are ready for field testing

Methods for COD and nitrate are being optimised

# Acknowledgements



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