# Next Generation Autonomous Chemical Sensors for Environmental Monitoring

# QUESTOR C E N T R E





## Deirdre Cogan, John Cleary, Thomas Phelan and Dermot Diamond

## **ENVIRON 2013**

#### Bringing Information to Life

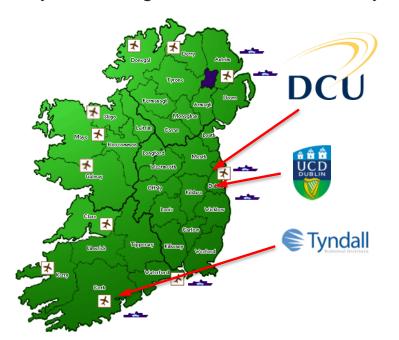
# SENSOR WEB TECHNOLOGIES

science foundation ireland fondurenth eolo-cite areann WWW.Clarity-Centre.org

Adaptive Sensors Group

1/

www.dcu.ie/chemistry/asg/





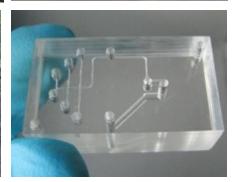








### Water sensing







## **Project Summary**

- 1. To produce environmental autonomous chemical sensing platforms with a price capability that creates a significant impact on existing market
- 2. Focusing on a detection platform for nutrients i.e. nitrite, nitrate and ammonia
- 3. Reducing price by integrating material chemistry into system
  - $\rightarrow$  Enable multiple targets to be detected using a single unit.

### $\rightarrow$ *Financial Benefit;*

*Futuristic biomimetic platform; tipping point in terms of scale of deployments for water quality monitoring* 

 $\rightarrow$  Significant market demand for low cost nutrient monitoring solutions for water and wastewater applications.

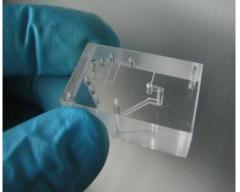
ENVIRON: 31<sup>st</sup> Jan 2013

#### Adaptive Sensors Group

## Microfluidics and Colorimetric Chemistry

### **Combination of Technologies**

- Colorimetric chemical assays
- Microfluidic systems
- Low cost LED/photodiodebased optical detection systems
- Wireless communications.

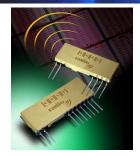




Ammonia

**Phosphate** 

 Developing low cost systems which can be deployed for extended periods providing high-frequency data on key water quality parameters.



#### Adaptive Sensors Group



Adaptive Sensors Group

## Phosphate Deployments

- Following an initial laboratory based calibration; the system was placed in situ at Broadmeadow Water Estuary, Co. Dublin for the period 22 Feb 2012 – 2 Mar 2012.
- Deployment of two systems in the Integrated Constructed Wetlands in Glaslough, Co. Monaghan from May – June 2012.



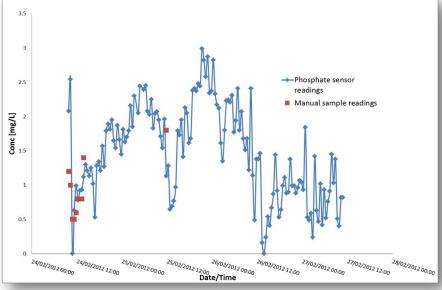
Adaptive Sensors Group

## Phosphate Deployment

### Broadmeadow 22Feb 2012- 2March 2012







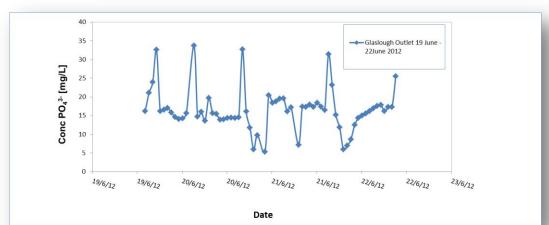
### Glaslough 22May-22June2012

 $\bigcirc$ 



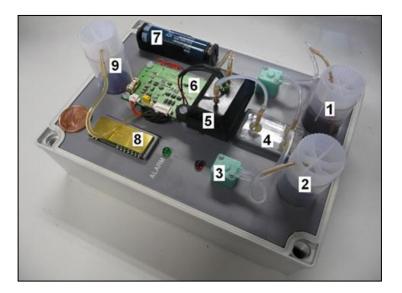
Adaptive Sensors Group





## **Determination of Nitrite - Griess Reagent**

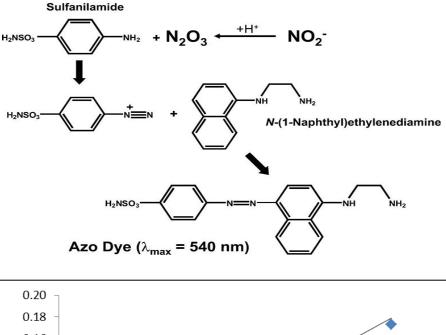


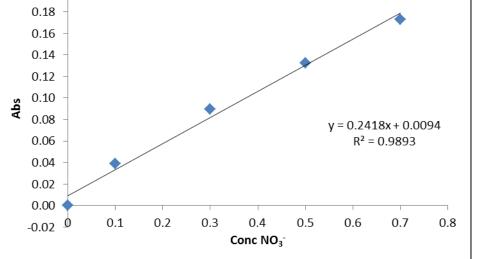


Benchtop nitrite detection system.

 $\bigcirc$ 

 $\bigcirc$ 



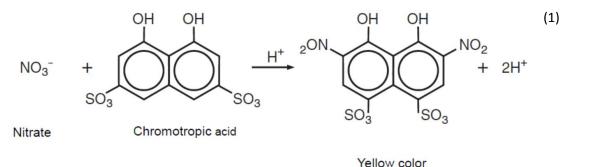


Adaptive Sensors Group

# **Determination of Nitrate**

- Current market for nitrate sensors use direct UV spectrophotometric screening, electrodes or cadmium reduction method which in turn can be quite costly.
- Major market for direct, inexpensive and robust sensor.

Determination of nitrate was investigated by a spectrophotometric method based on chromotropic acid. A yellow colour is developed when nitrate is treated with chromotropic acid in the presence of concentrated sulphuric acid and the absorbance measured at a wavelength of 430nm.





ENVIRON: 31st Jan 2013

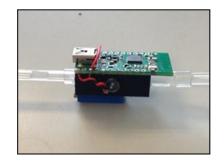
The work represented here on the study for the determination of nitrate has demonstrated that is possible to detect nitrate by a direct colorimetric technique.

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

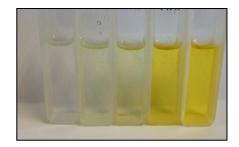
Adaptive Sensors Group

## Determination of Nitrate- Chromotropic Method





PEDD set up at 430nm

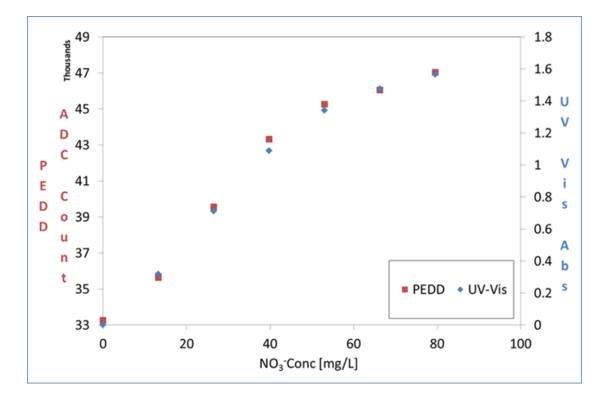


 $\mathbf{O}$ 

 $\mathbf{O}$ 

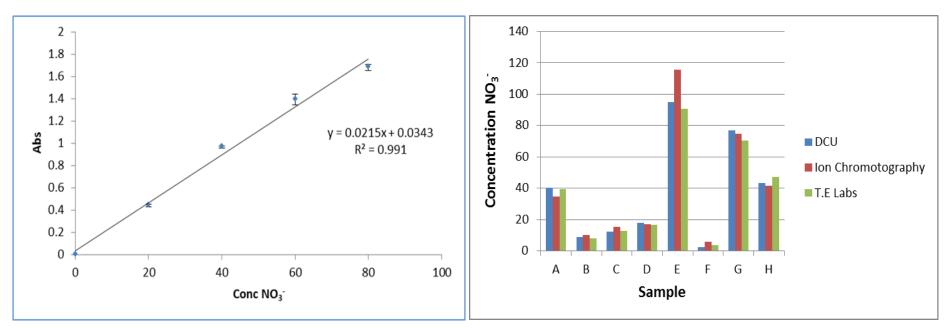
 $\mathbf{O}$ 

**Adaptive Sensors Group** 



) 🔾 🔾 🔾 🔾 💭 💭 ENVIRC

## Chromotropic Method vs. Ion Chromatography



#### Calibration Curve 0-80mg/L Nitrate

 $\mathbf{O}$ 

 $\mathbf{O}$ 

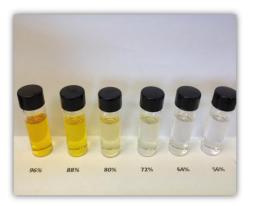
**Real Sample Blind Test** 

**Adaptive Sensors Group** 

# **Further Improvements**

### Improvements on Method: Decrease Concentration of Sulphuric Acid

Proved possible to achieve satisfactory analytical results



↓HEAT↓

0	-	-	-	-	
2	y	B			
96%	88%	80%	72%	64%	56%

 $\bigcirc$ 

 $\bigcirc$ 

 $\mathbf{O}$ 

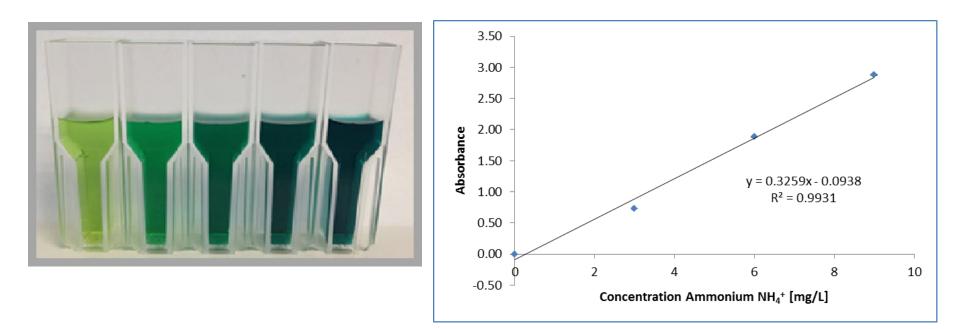
H <sub>2</sub> SO <sub>4</sub> Concentration	Result
96%	Immediate colour change when
88%	sample added to reagent.
80%	
72%	Resulted in a slight colour change
	when sample was added to
	reagent.
64%	No colour change when sample was
	added to reagent however after
56%	heating at a 130°C for 50mins, colour
	developed.

**RESULT:** Reducing risk factor while maximising lifetime of system

# **Determination of Ammonia**

The reagent cocktail includes a variation on:

- the Berthelot method which employs
- Salicylic acid instead of phenol which eliminates a toxic and relatively unstable reagent component.
- Intense colour generated is detected at a wavelength of 630nm.



# **Further Improvements**

- Previous method employs a three step reagent process
- Currently employing a mixed 2 step reagent process
- $\rightarrow$  Osberstown Waste Water Treatment Plant Sample = 0.6 mg/L NH<sub>4</sub><sup>+</sup>

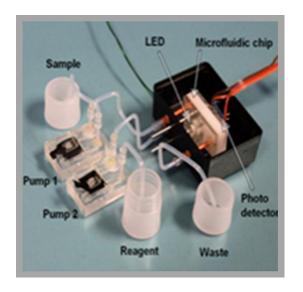
### → Using adjusted Berthelot Method:

Sample Type	Conc NH <sub>4</sub> <sup>+</sup> [mg/L]
Sample	0.6335
Sample + spiked with 1ppm	1.7416
Sample + spiked with 3ppm	3.6562
Sample + spiked with 5ppm	5.5696
HACH- Sample	0.75

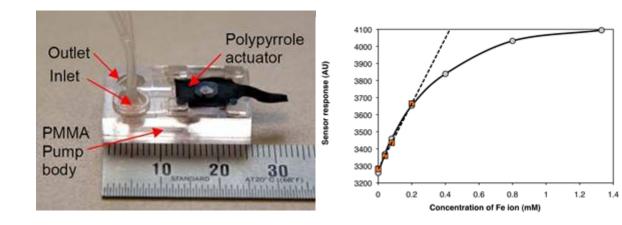
### **Future Work:**

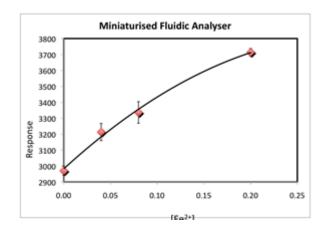
- Stability of reagents; hypochlorite
- Linear Range
- Integration into microfluidic platform following field deployment after appropriate lab testing

## Future Work- Generation 3; Futuristic Matchbox Analyser



- The goal is to integrate polymer actuator valves into the microfluidic chip, which will significantly drive down the overall cost of the platform.
- The main aims are to maximise the lifetime of these actuators through the use of ionic liquid electrolytes and optimisation of the pumping system (actuator stress/strain). This will achieve a fully integrated 'matchbox' analyser ready for field deployment.





### 'From Ecosystem Function to Human Health'



ISHTIMES.C	nment ( Emigratio			# 🚺
Home & treland » Recommend @ Tweek & treshtmes.com - Last Upcase: Tuesoux State 'compliant' OL	Rento	Distance Dis	<ul> <li>Big Phil st the muck 27/09/201</li> <li>Grants o septic ta</li> </ul>	payment rate lo (24/12/2012 ops stoically thro and madding cro

CIARA KENN



#### **IRISHTIMES.COM** Tuesday, .

News Sport | Business | Comment | Life | Societ Ireland | World Today's epaper | Generation Emigration | Vide Home a Ireland a

B Share Recommend 60 Tweet 10 Z +1 0 Irishtimes.com - Last Updated: Monday, August 6, 2012, 19:21

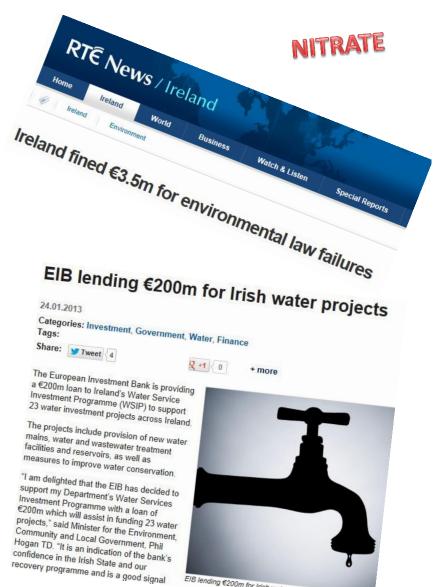
#### North Dublin water alert issued



OLIVIA KELLY

Residents of up to 1,400 houses in north Dublin have been warned not to c high levels of bacteria in the system.

Dublin City Council has issued a "major alert" to the homes and properties



EIB lending €200m for Irish water projects

#### Adaptive Sensors Group

#### $\bigcirc$ $\bigcirc$ $\bigcirc$

# Thank you for your attention

We acknowledge support for this research from The Questor Centre (grant code DCU9/11/14).



