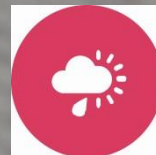




UNIVERSITY
OF HULL

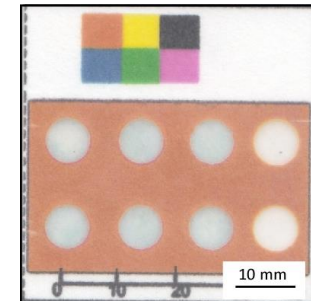
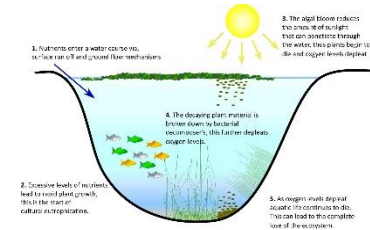
Paper-Based Devices for Environmental Analysis

Samantha Richardson,
Alexander Iles,
Jeanette Rotchell, Mark Lorch,
Nicole Pamme



Sustainable North Sea Region
<http://northsearegion.eu/sullied-sediments>

- i. Background
- ii. Device fabrication and experimental procedure
- iii. PADs for phosphate detection
- iv. Volunteer sampling campaign





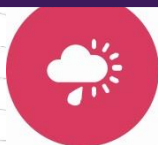
Sullied Sediments project

- better assessment
- better treatment
- better prevention of contamination

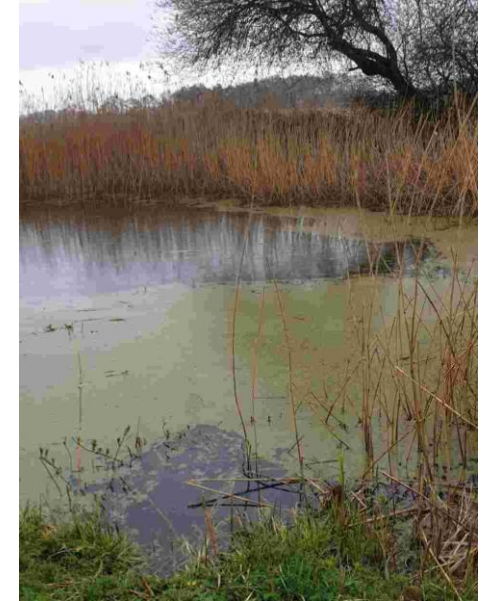
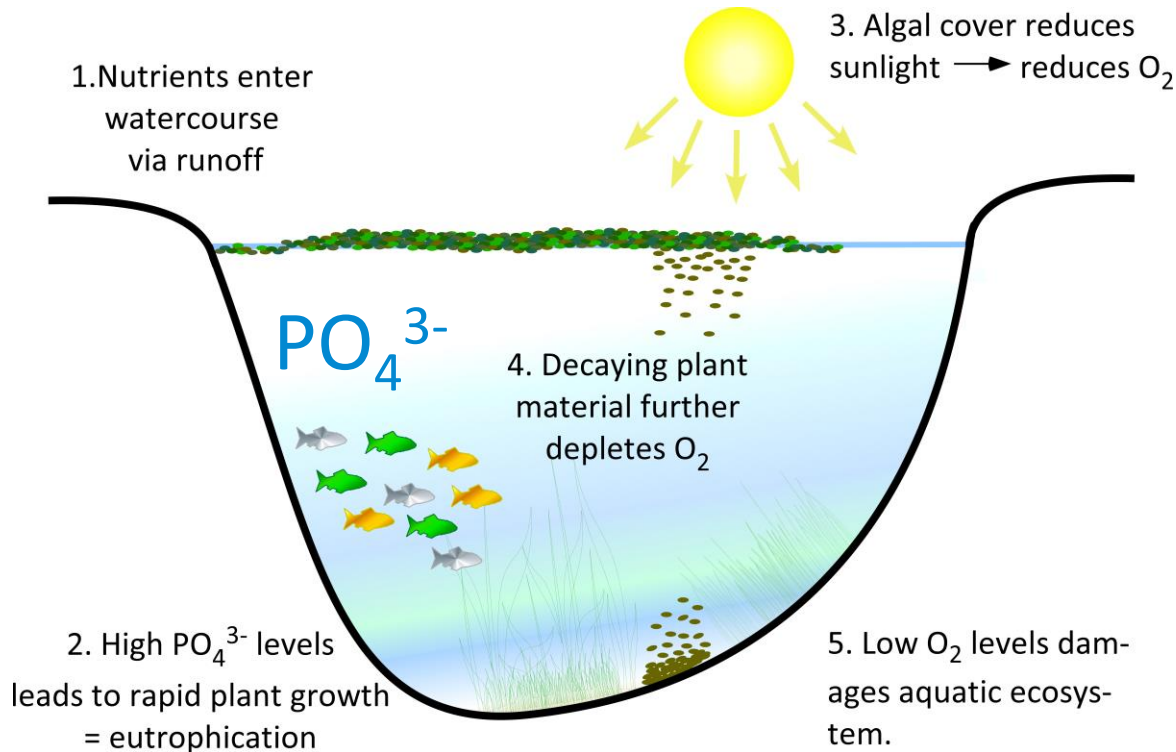
river catchment partners:

- UK (Humber)
- Germany (Elbe)
- Belgium / NL (Scheldt)

members of the public to measure pollutants in river/canal water
→ Dipstick Test as an engagement tool



river pollution (PO_4^{3-})



$\text{PO}_4^{3-} > 0.1 \text{ mg L}^{-1}$
→ eutrophication

- Reactive phosphate in aquatic systems key nutrient for eutrophication
- Orthophosphates main bioavailable phosphate in aquatic systems

conventional analysis

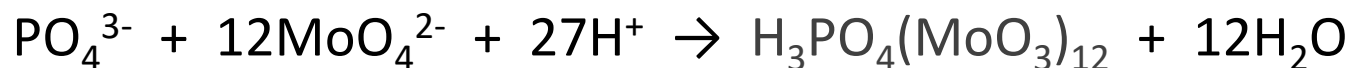
Laboratory-based analysis

- Reaction to form blue complex
- UV-vis spectrometry readout (£100s - £1000s)
- Expert analyst
- Time intensive (hours – days)

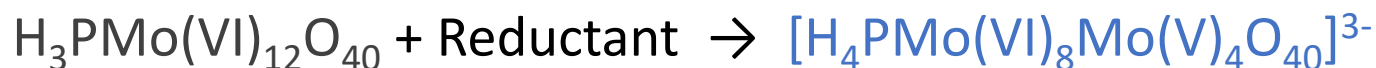


Phosphomolybdenum blue (PMB) reaction

Step 1

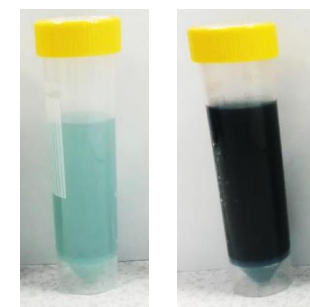


Step 2



Colourless

Blue



low
[PO₄³⁻]

high
[PO₄³⁻]

Requires pH<1 → achieved using sulfuric acid

conventional analysis

Laboratory-based analysis

- Reaction to form blue complex
- UV-vis spectrometry readout (£100s - £1000s)
- Expert analyst
- Time intensive (hours – days)



Field-based analysis

- Handheld photometer
- Equipment and consumable cost (£100s)
- Expert analyst
- Time consuming
(calibration for individual analyte)



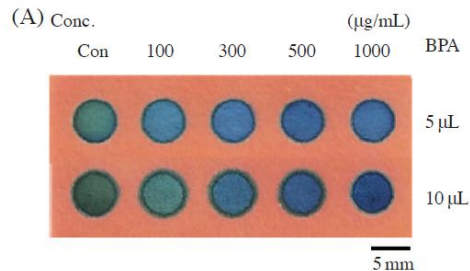
Hach Pocket Colorimeter
<https://catchmentbasedapproach.org/learn/citizen-science-equipment-list/>

Desirable	→ Simple operation (layperson), use in-the-field
	→ Quick result (few min) and inexpensive (<£1)

paper-based analytical devices (PADs)

Spot tests

bisphenol A



Easy to
understand
colour readout

requires
pipetting

Park *et al.*,
BioChip J., **2016**, 10, 25.

Lateral flow

explosives residues



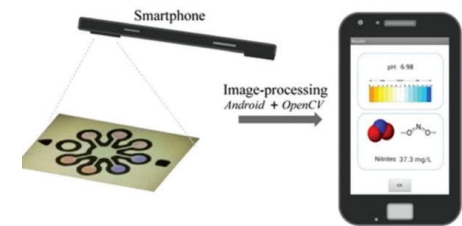
Potential for
multiplex
analysis

long time
to readout

Peters *et al.*,
Anal. Meth., **2015**, 7, 63.

Smartphone capture

pH / nitrite

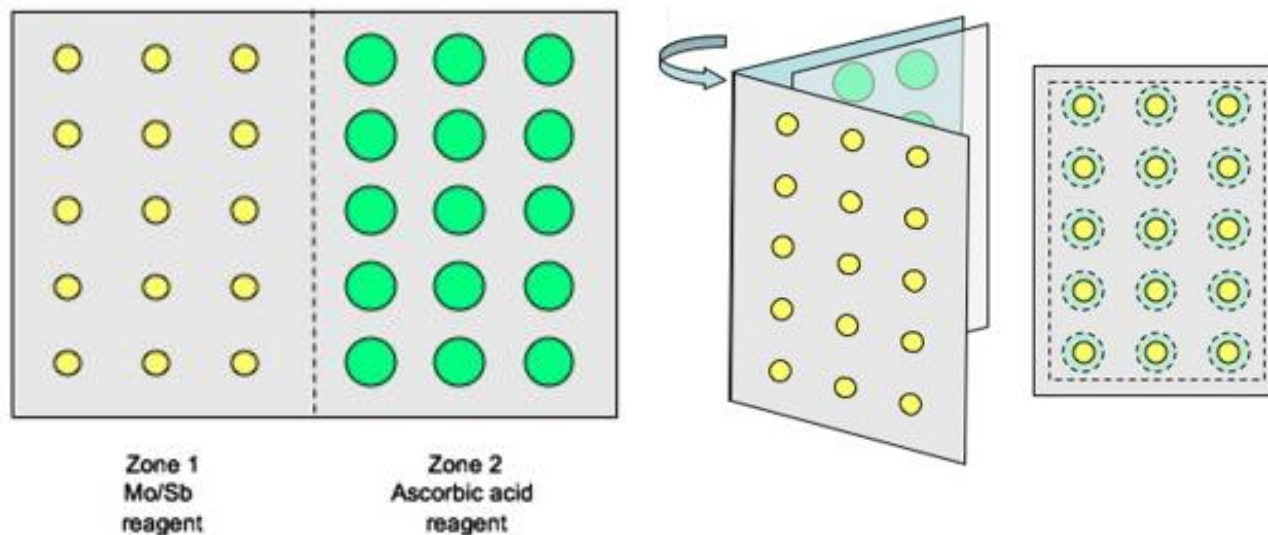


Ideal detector
for in-field
result capture

data
processing

Lopez-Ruiz *et al.*,
Anal. Chem., **2014**, 86, 19, 9554.

PAD for phosphate detection



PMB reaction

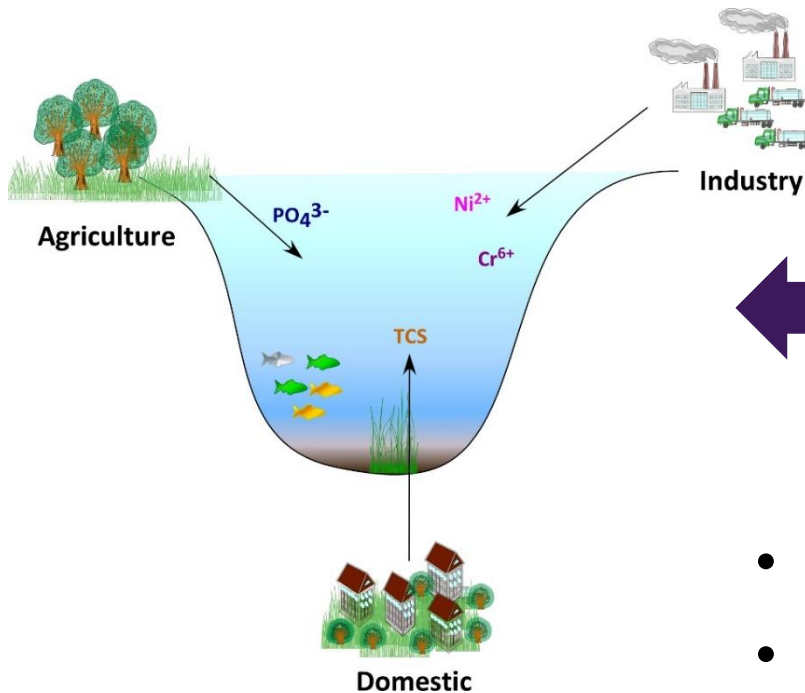
- 2 step reaction (2 zones)
- pH < 1 (6.6 M H₂SO₄)
- Reagent stability (Teflon sheet)

- Multistep workflow
- Pipetting required
- Takes up to 40 min
- Sulfuric acid degrades cellulose over time
- Scanner used for image capture

Unsuitable for
our envisaged
volunteer field
sampling

our desired workflow

Regular monitoring of pollution pressures

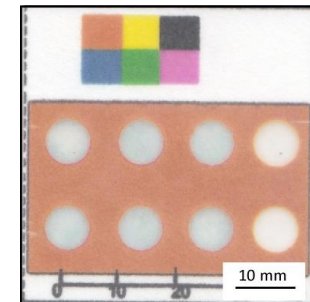
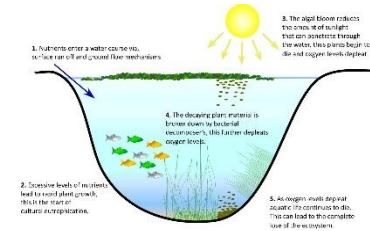


Dip test

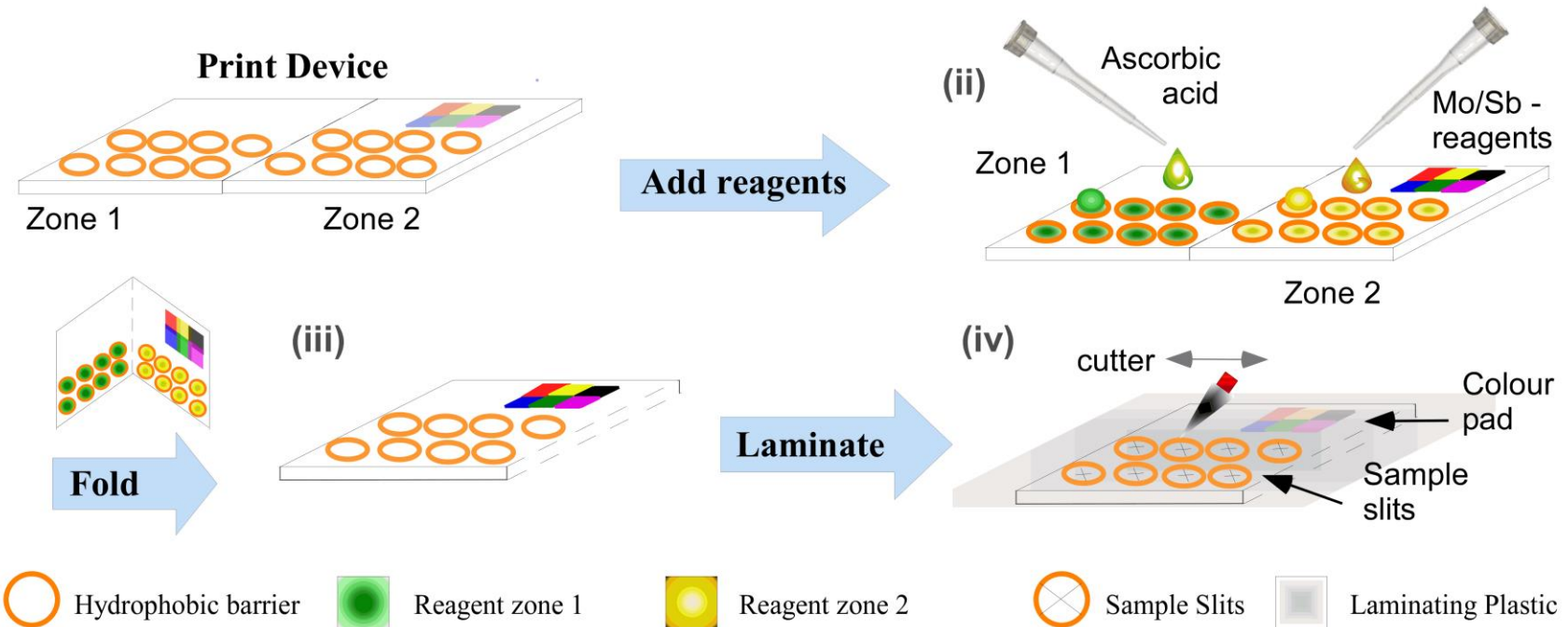


- Interactive tool (engagement)
- Simple (volunteers)
- Use in the field (quick, minimal input)

- i. Background
- ii. Device fabrication and experimental procedure
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- iv. Volunteer sampling campaign



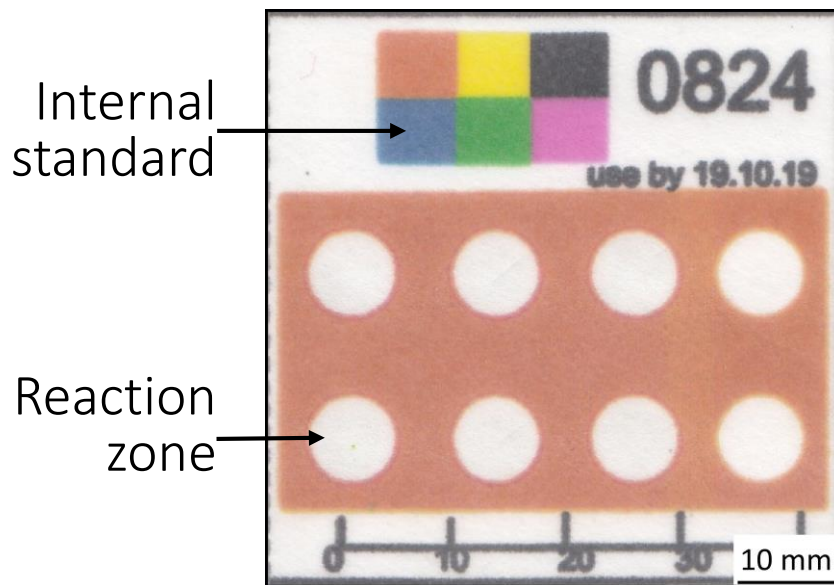
device preparation



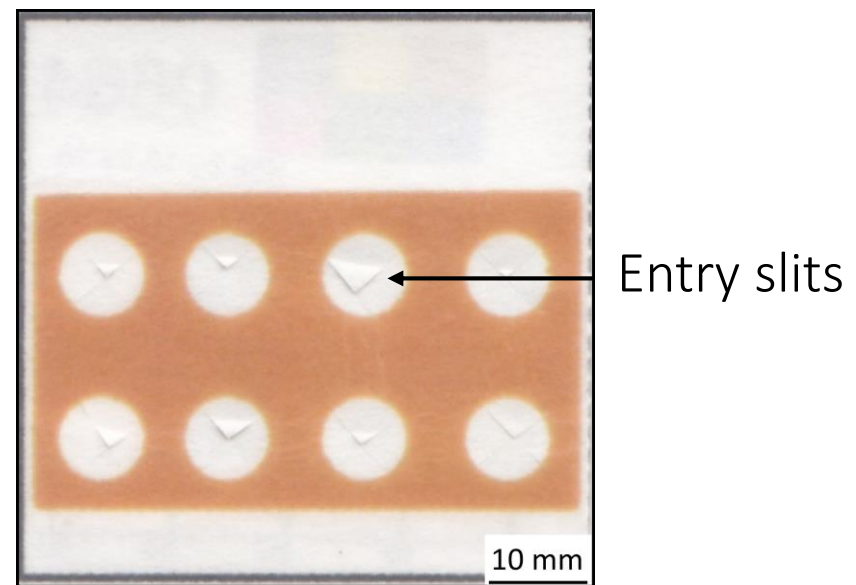
- Aggressive sulfuric acid [6.6 M] replaced p-toluene-sulfonic acid [2 M]
- Auto-reduction minimised by reducing [molybdenum] 10x
- Reagents spatially separated via back-folding to allow for two sequential reaction steps
- Reaction zones 10 mm in diameter for easy readout

fully prepared devices

Front of device



Back of device

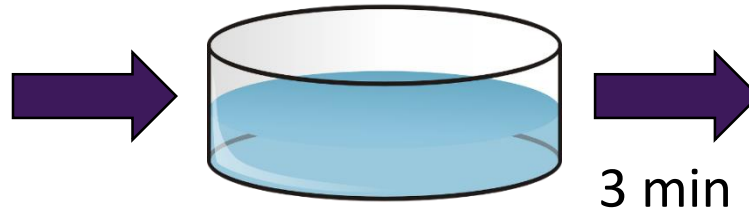


user workflow

1. Collect water sample



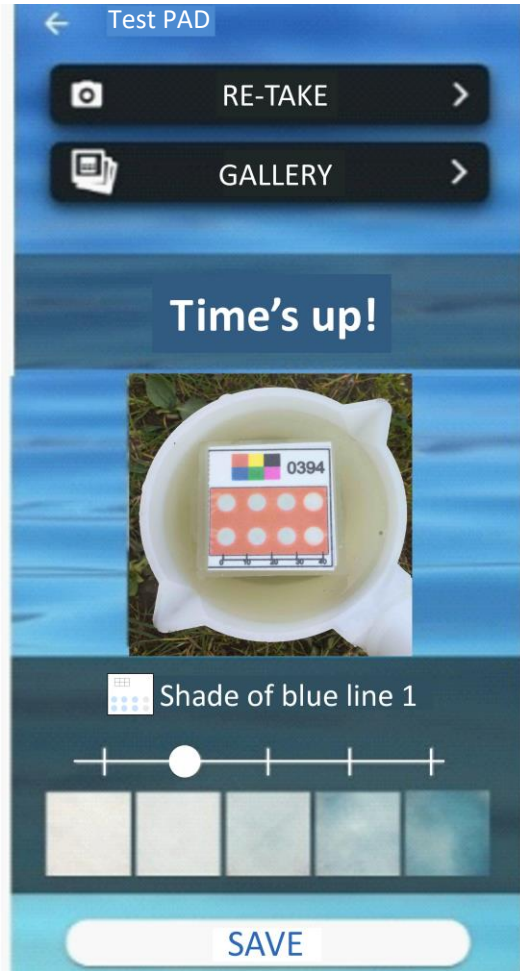
2. Place small amount of river water in container and add test pad



3. Take photo and upload to app



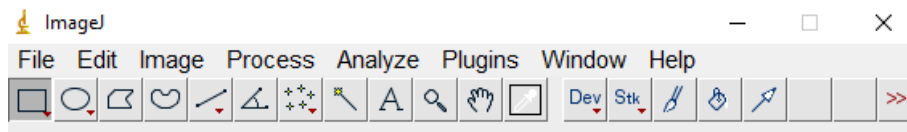
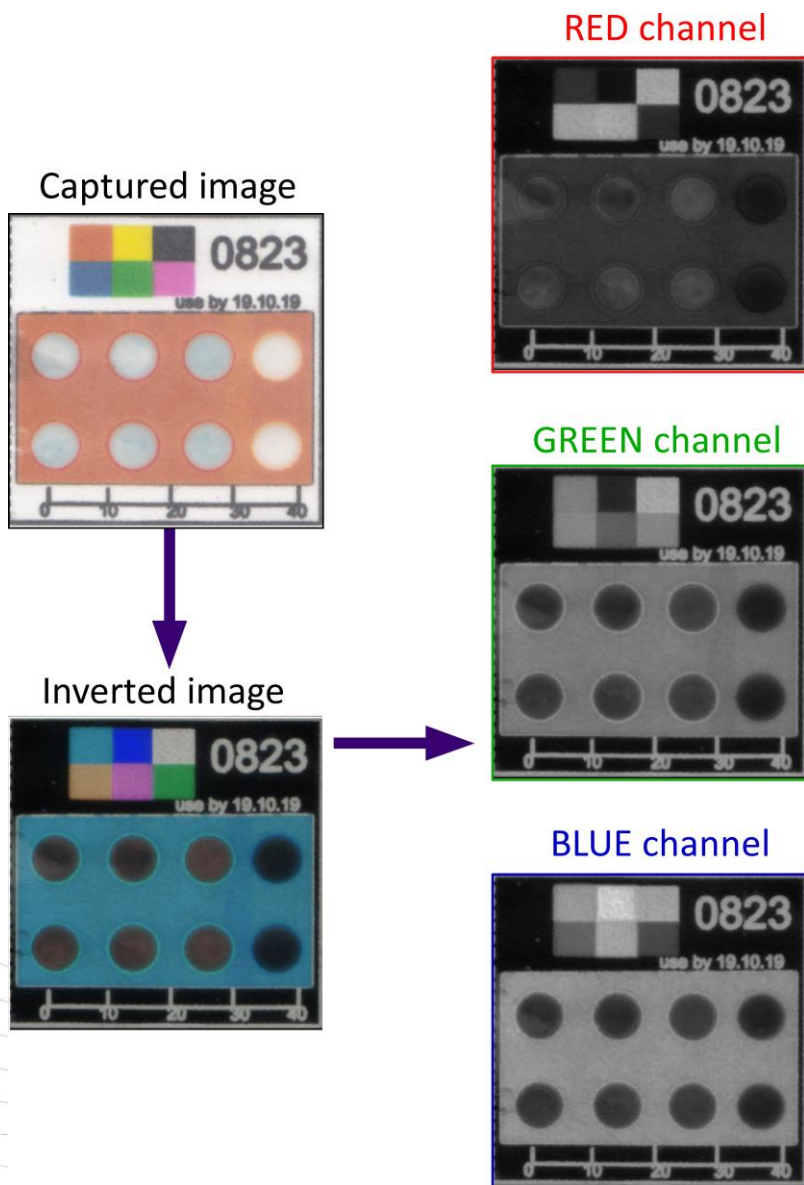
custom-designed RiverDip app



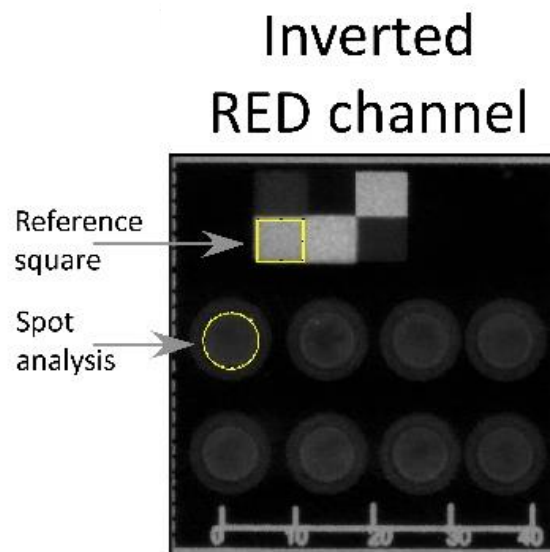
- Photograph following 3 min timer
- User can self-interpret intensity on scale
- GPS data, time
- PAD id code entered
- User also record photograph of landscape and water colour

Data held on sever for further analysis

image analysis

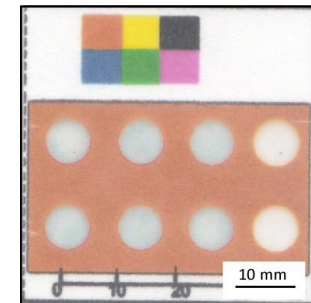
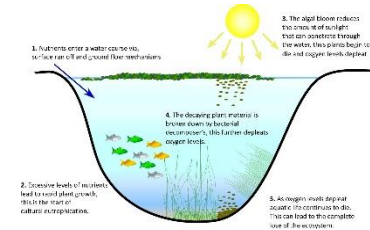


<https://imagej.nih.gov/ij/>



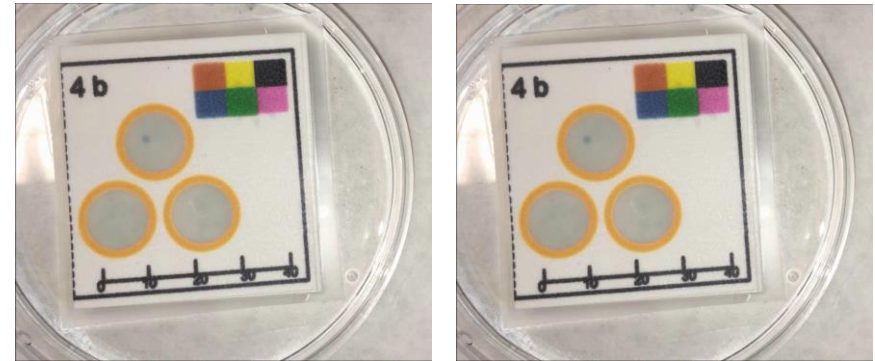
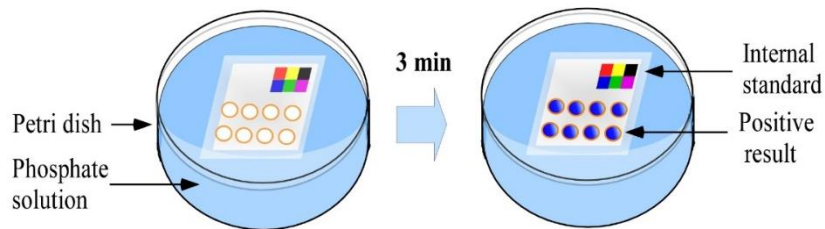
measured average pixel intensity

- i. Background
- ii. Device fabrication and experimental procedure
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- iv. Volunteer sampling campaign

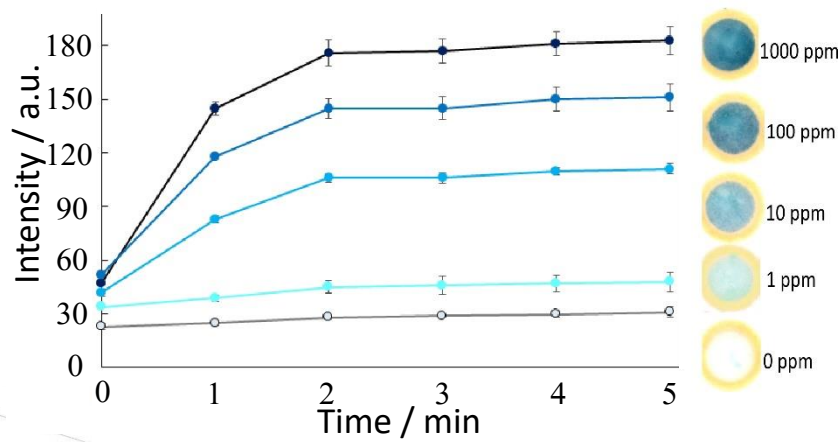


lab-based development

PO_4^{3-} spiked into purified water
(0, 10, 100, 1,000 ppm)



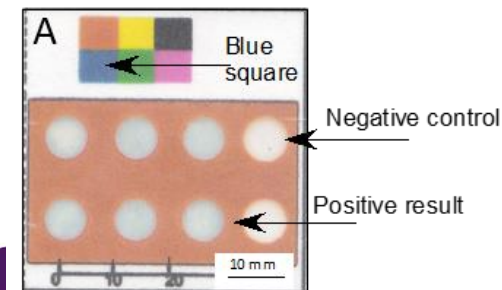
Optimising incubation time



3 min = optimum time

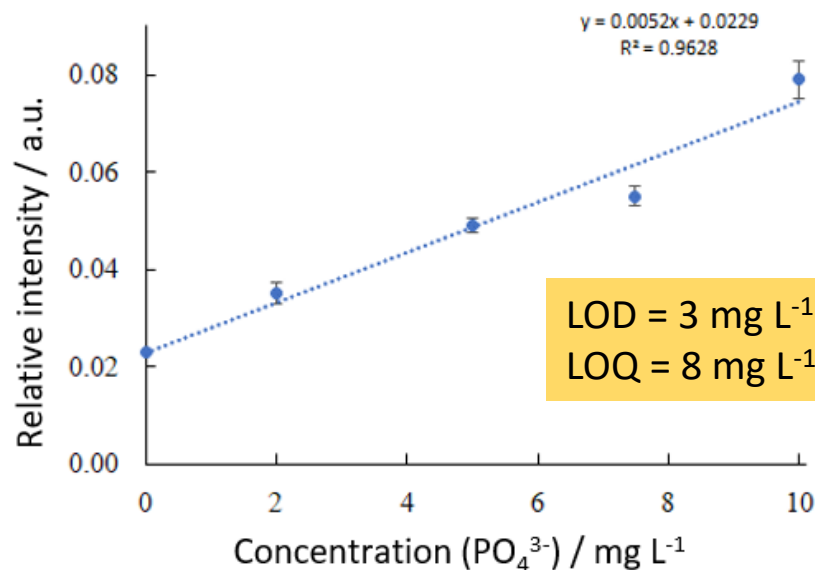
Tested for interferences

- Silicates found to not affect reading in 3 min timescale
- Any water colour accounted for in reference circle



development of field devices

Calibration curve for determining PO_4^{3-} in the field

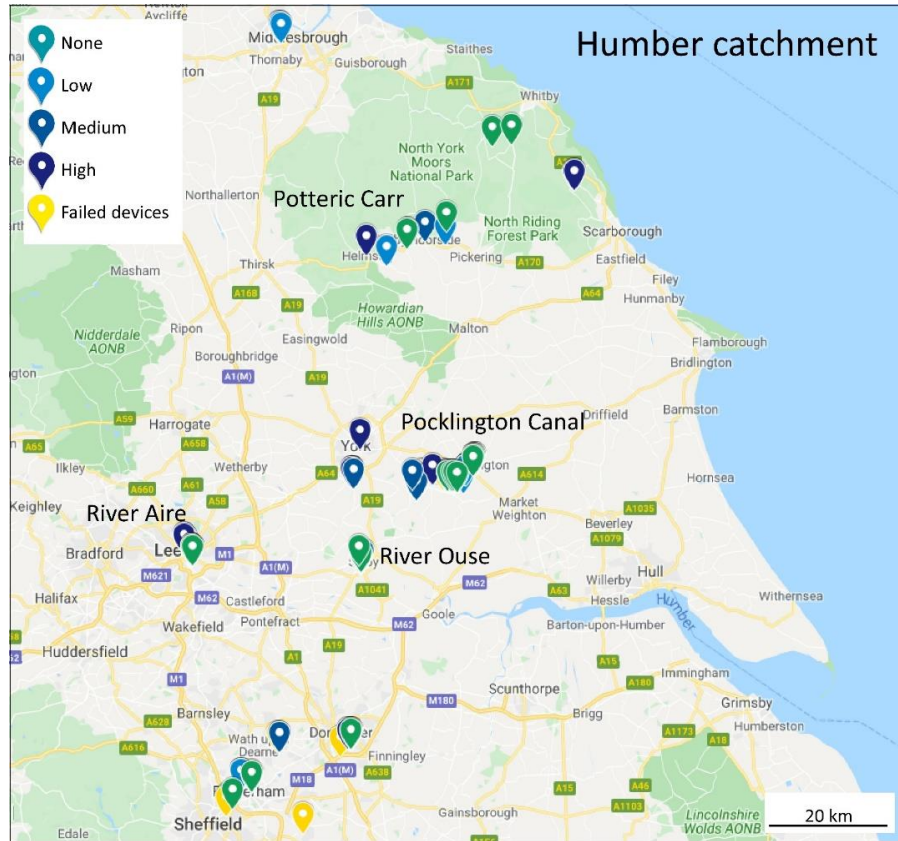


Benchmarking

Water sample	UV/VIS	PAD
	Average PO_4^{3-} / mg L^{-1}	Average PO_4^{3-} / mg L^{-1}
River Aire 1	0.4 ± 0.006	0.3 ± 0.020
River Aire 2	2.9 ± 0.040	3.1 ± 0.020
River Aire 3	2.1 ± 0.100	1.8 ± 0.100
Pocklington Canal	0.0 ± 0.001	0.1 ± 0.009

at environmental levels
($\text{PO}_4^{3-} = 0.1 - 10 \text{ mg L}^{-1}$)
→ PADs and UV/vis method
give comparable results

volunteer campaign



→ >100 tests carried out so far

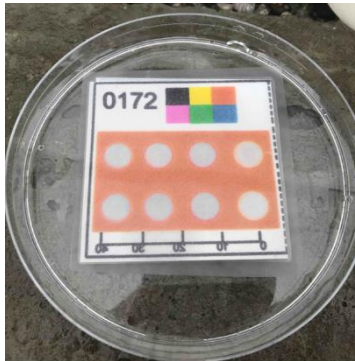
Humber catchment, UK
Led by Canal & Rivers Trust
(local managers of waterways)

Key challenges

- Getting volunteers to use app
- Image capture in the field



example – Pocklington Canal

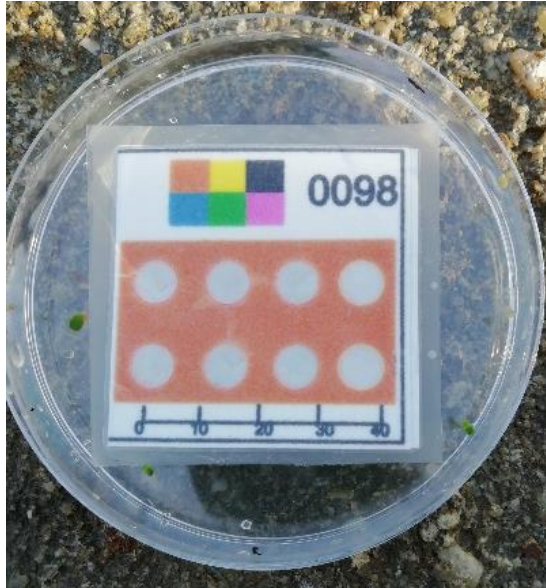


<https://www.google.com/maps/d/u/0/viewer?hl=en&h=en&mid=1b-f0thmOKrGvix8XCom7VgdPh89b6e&ll=52.8530205131834742C1.142253478500038&z=7>

Repeated sampling led by PCAS volunteers
across 7 km stretch of canal (April, June, September)

→ Patterns emerge across different sampling periods

conclusions



- Developed simple and ‘instrument-free’ paper analytical devices (PADs) for environmental analysis have been developed.
- Demonstrated workflow and data capture methods suitable for field use.
- PADs for phosphate detection successfully tested by volunteers, being rolled out across North Sea Region.
- Developing PADs for other environmental pollutants.

acknowledge- ments

Volunteer organisations

- Canal & Rivers Trust
- Pocklington Canal Amenity Society



RiverDIP app development

Natural aptitude



Funders

Interreg North Sea region European
Regional Development



Sustainable North Sea Region
<http://northsearegion.eu/sullied-sediments>