Politecnico di Torino
Technion Institute of Technology
University of Torino
EXPO 2015

Managing Water Quality for Public Health

Department of Agriculture, Forest and Food Sciences University of Torino

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Autonomous system design for continuous monitoring of metals in water

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Materials and Processes for Micro & Nano Technologies

POLITECNICO DI TORINO

http://www.polito.it/micronanotech

http://www.chilab.polito.it

Prof. Fabrizio Pirri

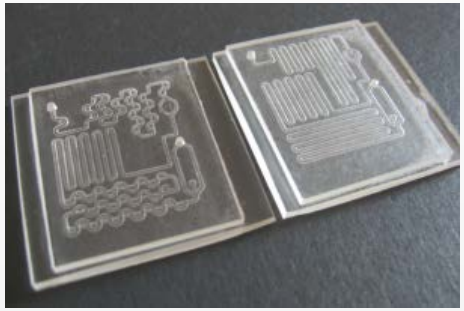
Research Leader

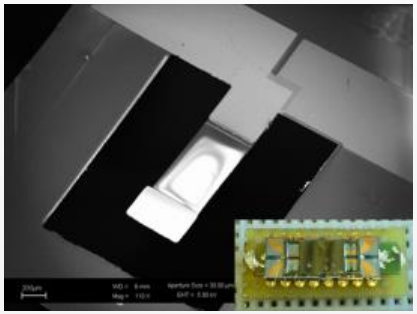
Staff

- 5 Professors
- 8 Permanent Researchers
- 16 Fellowships / Post Doc
- 8 PhD students
- 3 Technicians
- 2 Administratives

Mission

- fundamental research on materials and processes for micro- and nano-technologies
- design and fabrication of MEMS and nanostructures
- technological transfer
- education





Out line

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1. Spectrophotometry techniques
2. Calibration curves Spectrophotometry technique
3. Design and engineering of a portable instrument
4. Waste water monitoring
5. Fresh water monitoring
6. AUV payload engineering
7. Results and Future developments

Spectrophotometry techniques

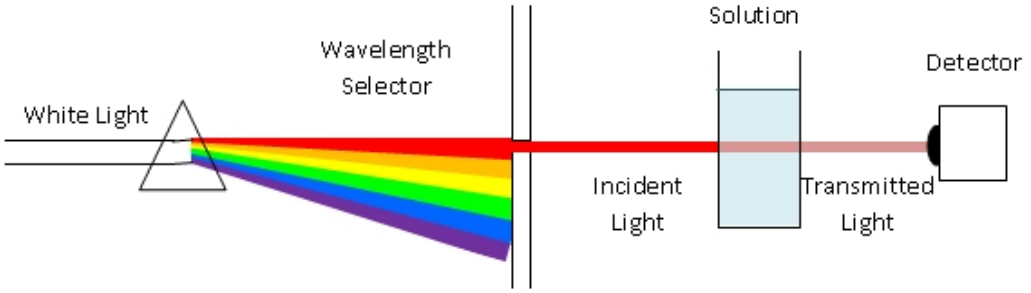
Several techniques such as X-ray fluorescence, atomic fluorescence spectrometry, chromatography, atomic absorption spectrometry, etc. have been used for the simultaneous determination of different ions in different samples.

Among the most widely used analytical methods are those based on the **UV-Vis spectrophotometry techniques**, due to the resulting *experimental rapidity*, *simplicity* and the *wide application*.

Nowadays quantitative spectrophotometry has been greatly improved by the use of a variety of **multivariate statistical method**; particularly principle component regression (PCR) and **partial least squares regression** (**PLS**). PLS regression has been found important in handling regression tasks in case there are many variables. **PLS allows to simultaneous determination of different ions in water compounds**.

Spectrophotometry techniques

Spectrophotometry is a method to measure how much a chemical substance absorbs light by measuring the intensity of light as a beam of light passes through sample solution. The basic principle is that each compound absorbs or transmits light over a certain range of wavelength.



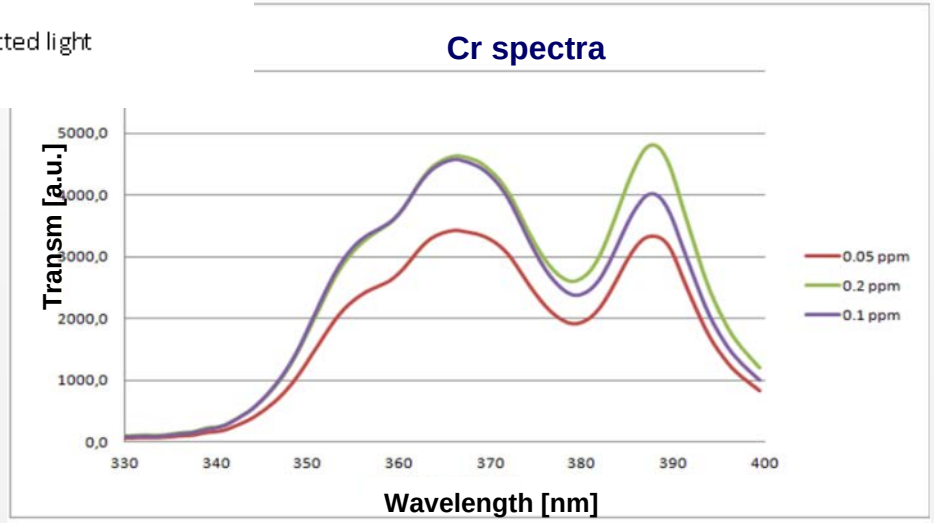
The difference between the incident and transmitted light indicates the absorbance

$\log I_0/I$ = Absorbance

Beer's Law

Absorbance = ε x conc x l

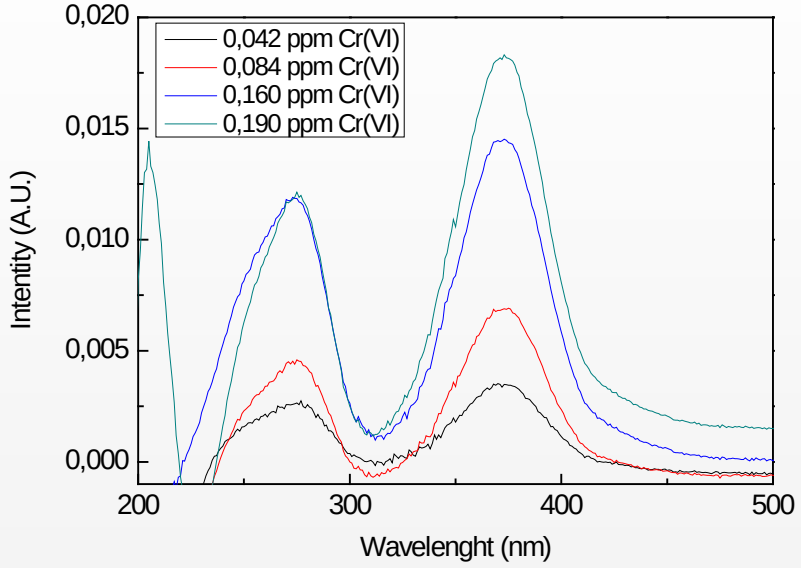
The adsorption spectra of three different Cr concentrations in drinking water has been compared. The finger print is obtained by subtracting the reference spectrum.

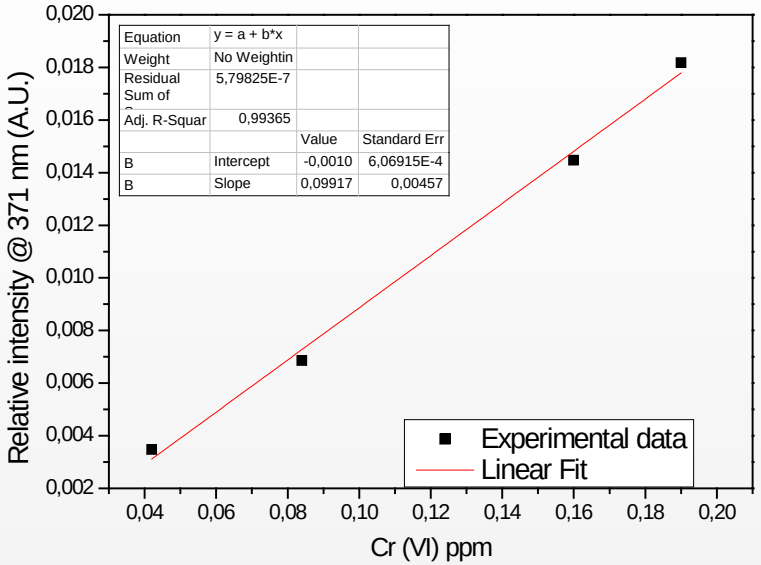


Calibration curves Spectrophotometry technique

Spectrophotometry technique applied for the measurement of Cr (VI) concentration in waste water

The quantitative analysis is obtained by considering the absorption peak at 371 nm.

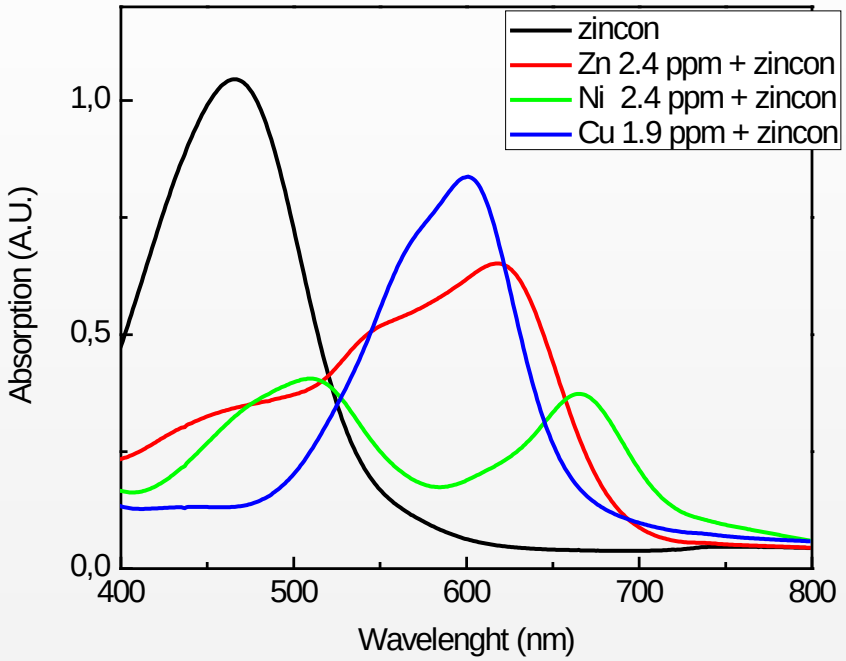




Quantitative determination of hexavalent chromium in aqueous solutions by UV-Vis spectrophotometer
M. C. Fournier–Salauun and P. Salauun
Central European Journal of Chemistry *5(4) 2007 1084–1093*

Calibration curves Spectrophotometry technique

The partial least squares (PLS) has been applied to the simultaneous determination of the divalent ions of **copper**, **nickel** and **zinc** based on the formation of their complexes with 2-carboxy-2-hydroxy-5-sulfoformazyl benzene (zincon). The absorption spectra were recorded in the visible spectrum (400 - 800 nm).



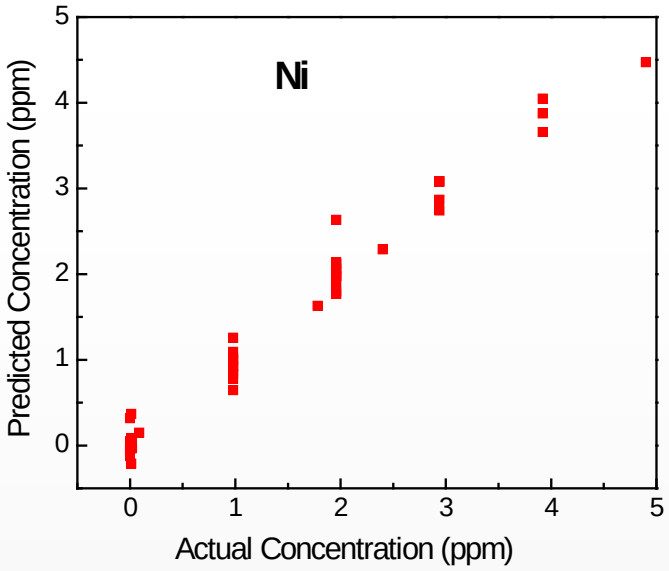
Spectra of different solution are different, nevertheless the superposition presence does not permit a direct measurement by means of absorption spectra.

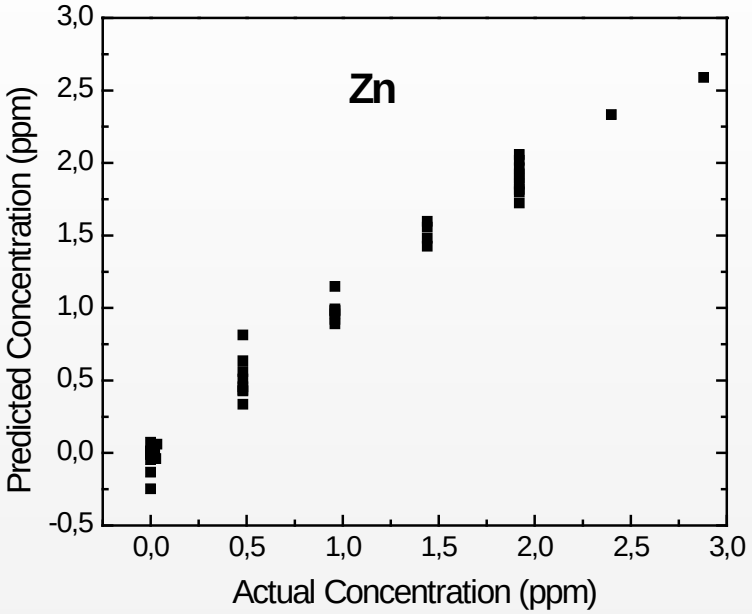
PLSR: partial least squares regression is required

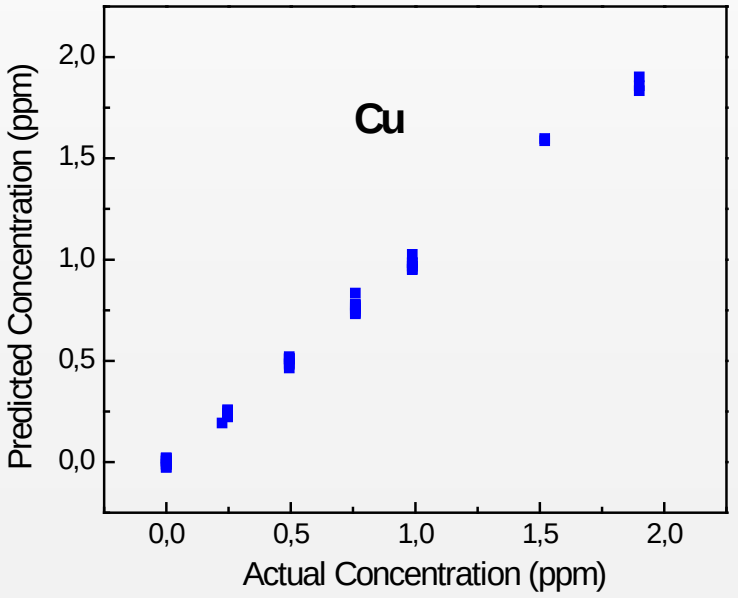
Simultaneous determination of copper, nickel, cobalt and zinc using zincon as a metallochromic indicator with partial least squares
J. Ghasemi, Sh. Ahmadi, K. Torkestani
Analytica Chimica Acta 487 (2003) 181–188

Calibration curves Spectrophotometry technique

Experimental results: Plots of predicted concentration vs. actual concentration for three cations (Copper, Nickel, Zinc) in the prediction set.







Calibration curves Spectrophotometry technique

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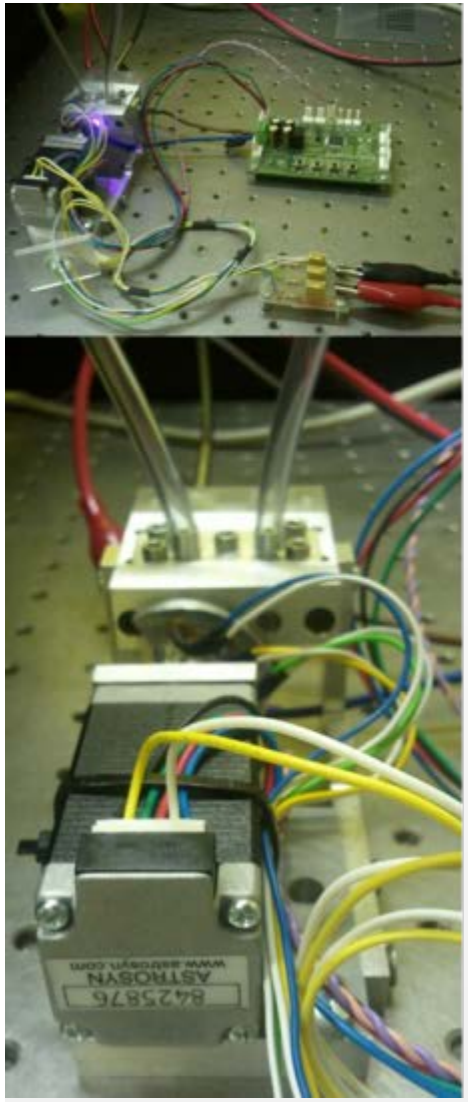
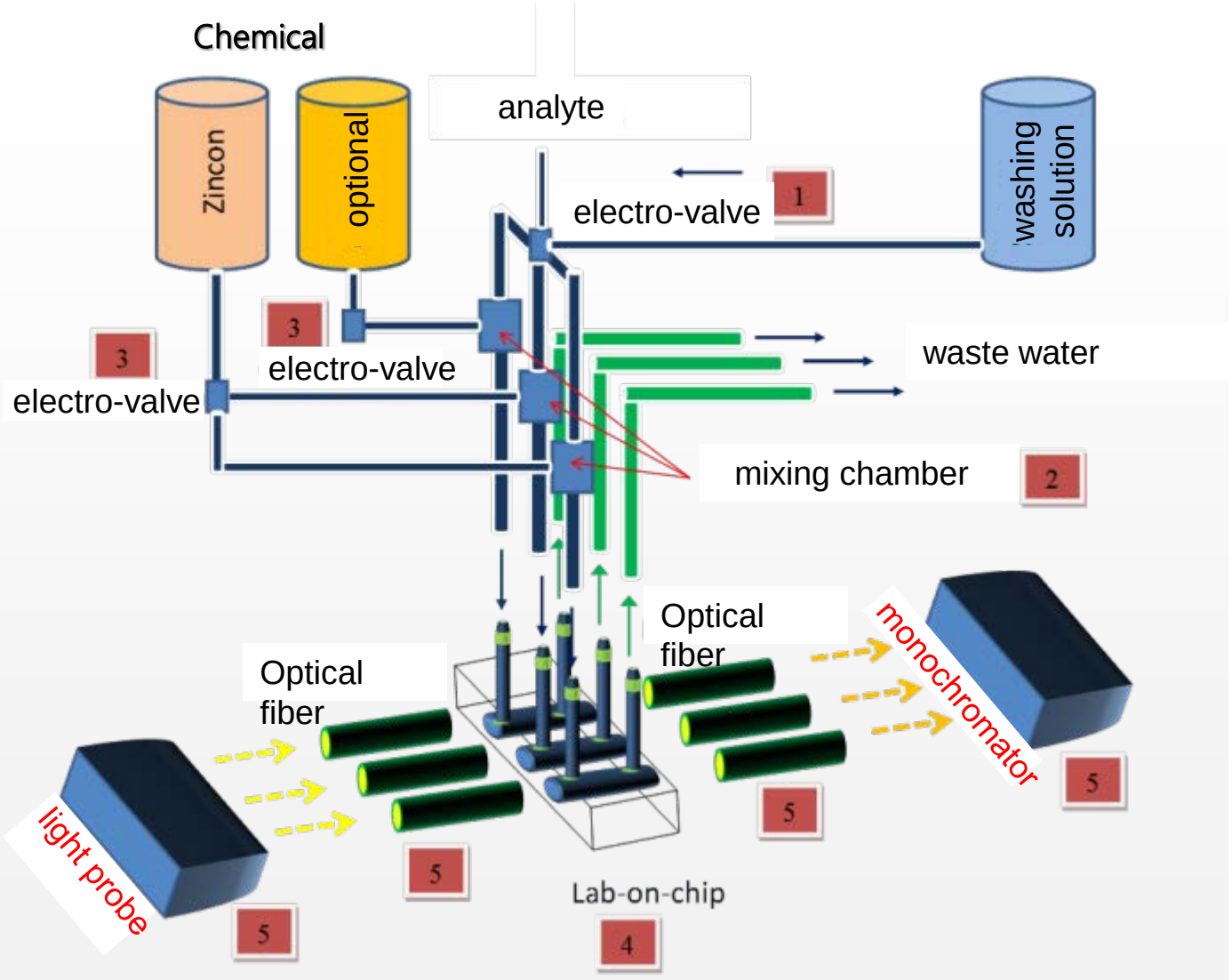
In order to test the reliability of the proposed method different tests has been performed in a variety of synthetic solutions. The results of the prediction are summarized in the table.

Sample	Zn (ppm)	Zn prediction (ppm)	Ni (ppm)	Ni prediction (ppm)	Cu (ppm)	Cu prediction (ppm)
Z5	0,25	0,22	2,9	3	0,96	0,98
Z25	0,76	0,75	1,9	1,8	1,9	1,8
Z36	1,9	1,8	0,9	0,7	1	1
12906-0,45um	0	0,01	0,01	0	0,02	0,05
12906-8ul	0	0,01	0,01	-0,2	0,02	0
12906-100ul	0	0,02	0,01	0,3	0,01	-0,01

Design and engineering of a portable instrument

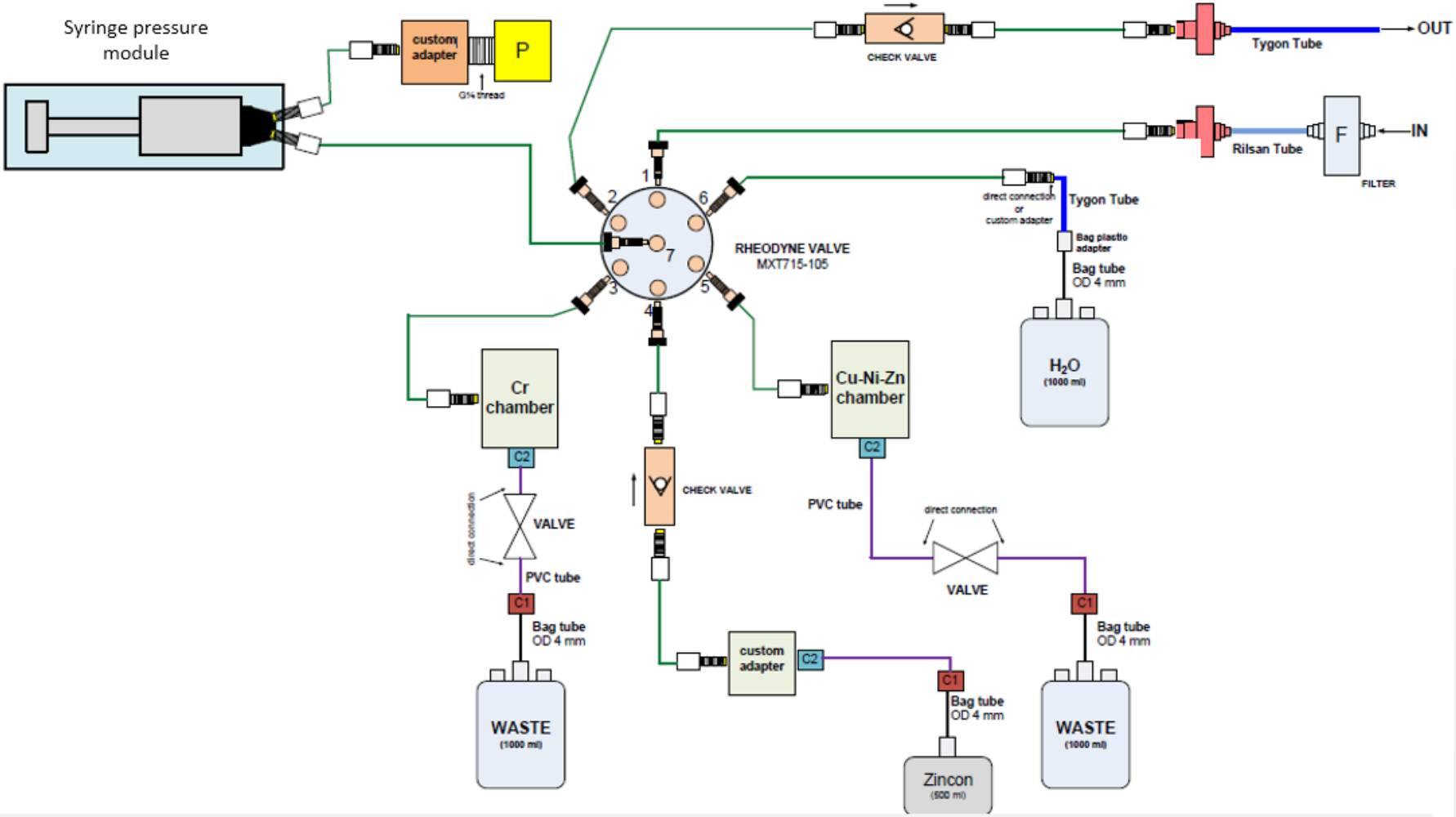
Functional schema and preliminary tests



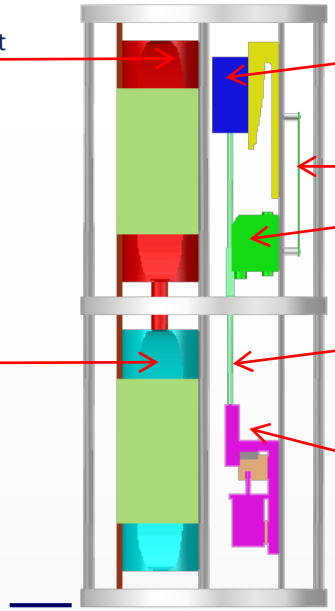


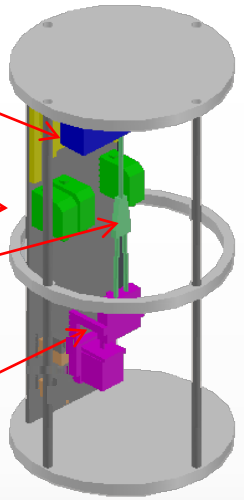
Design and engineering of a portable instrument

Assembly schema

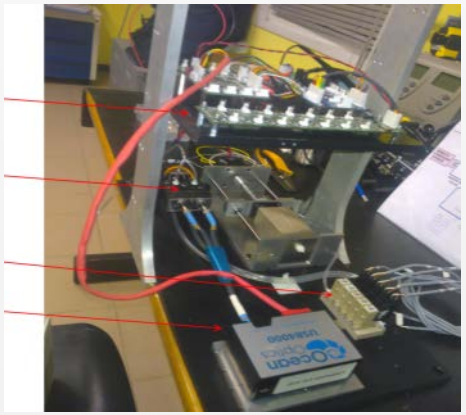


Design and engineering of a portable instrument





SMAT S.p.A.



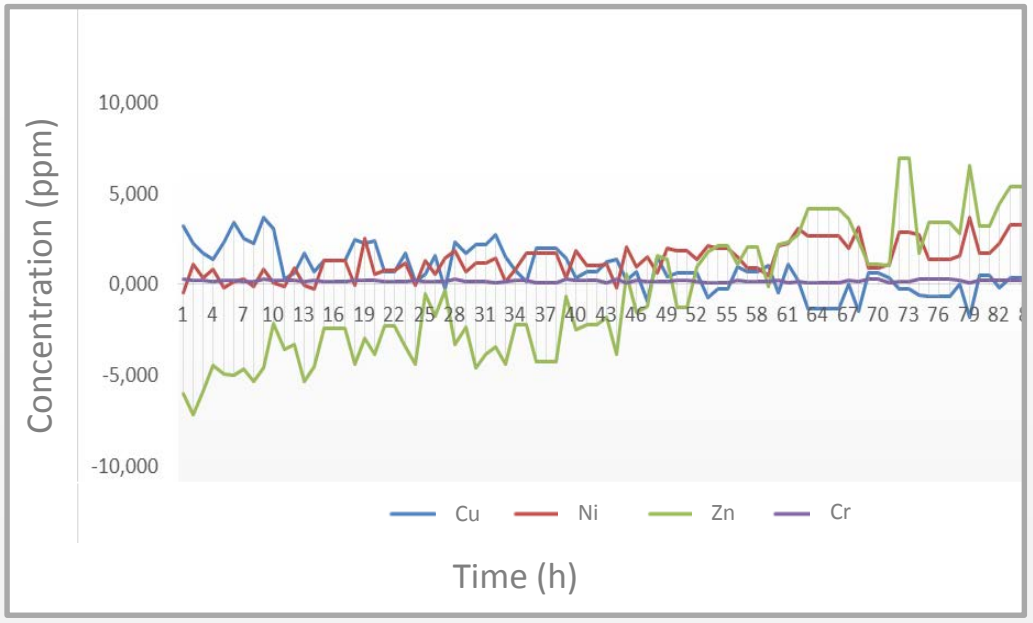
Eni S.p.A.

Waste water monitoring test – Castiglione T.se (TO) - Italy

WMA laboratory test

Ni, resolution	0.8	ppm
Cu, resolution	0.8	ppm
Cr VI, resolution	0.8	ppm
Zn, resolution	0.8	ppm
Measurement range	0 ÷ 10	ppm

WMA installed



Continuous autonomous monitoring – Preliminary data

Fresh water monitoring test – Torino - Italy

WMA installed

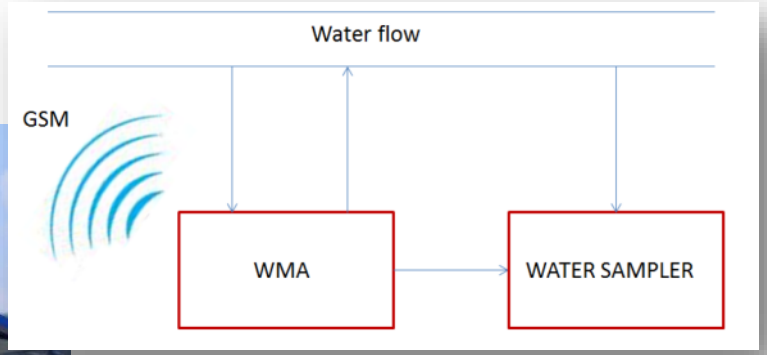
Fresh water SMAT's delivery point

- Test performed thanks to the cooperation with SMAT S.p.A. and the grant POR-FESR 2007/2013 by Piedmont Region

UNIONE EUROPEA

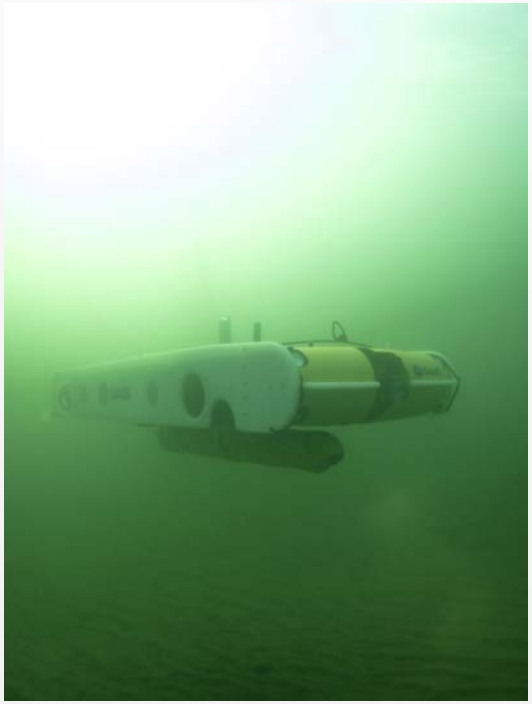
REGIONE PIEMONTE

- Continuous autonomous monitoring – Preliminary data
- Data collected by GSM



Measurement data sending

AUV payload engineering - test on VATTERN Lake, Motala –Sweden



AUV payload engineering - test on VATTERN Lake, Motala –Sweden

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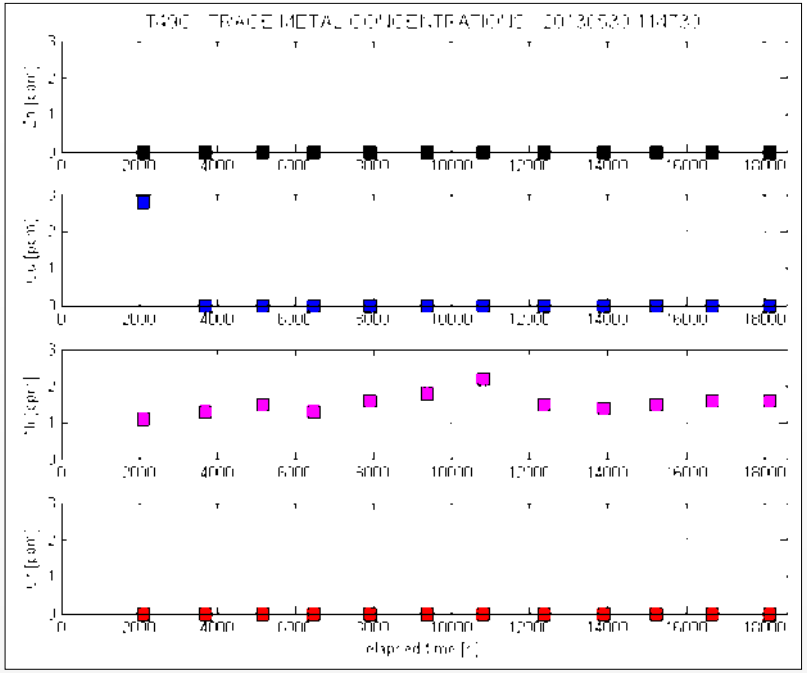
- E-POD#3 successfully operated
- Spectrophotometric technique demonstrated
- All functionalities verified, no failure occurred

- Bias on Ni observed during field tests, not present during calibration

- Laboratory analysis on water samples collected during the tests
  - confirmed in-situ measurements of Cu, Zn, Cr
  - anomaly of in-situ measure of Ni possibly due to interference of Zincon with organic substances (absorption in the same wavelength)

- Expansion to other parameters (further trace metals, hydrocarbons) possible

- method sensitivity presently 0.7 ppm, optimization required to reach 100 ppb target (best lab.technique)



Results and Future developments

- Prototype analyser developed, **laboratory calibrated** and **fields tested**
- Concept validated (TRL 2), **optimisation work required**
- A possible qualification program (target TRL 4) should include
    - System design review; study possible optimisation of the system layout and components (and opportunity to add new parameters)
    - Laboratory tests to optimise analytical cycles and explore the limits of the system (sensitivity, repeatability, long-term duration, pressure, temperature, long-term etc.)
    - Test in a real environment (e.g. characterised by industrial pollution)

Thank you for the attention

Particular thanks to:

- Paola Rivolo, PhD
- Alessandro Virga, PhD

Thanks to:

SMAT S.p.A.

Eni S.p.A.

Microla Optoelectronics S.r.l. for the engineering of the system.