Field testing, validation and optimization report

Deliverable D9.2
of the COMMON SENSE project

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Deliverable 9.2

Field testing, validation and optimization report

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COMMON SENSE
MARINE SENSORS - MARINE MONITORING

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EXECUTIVE SUMMARY

The COMMON SENSE project has been designed and planned in order to meet the general and specific scientific and technical objectives mentioned in its Description of Work (page 77).

As the overall strategy, the 11 work packages (WPs) of the work plan were grouped into 3 key phases: (1) RD basis for cost-effective sensor development , (2) Sensor development, sensor web platform and integration, and (3) Field testing. In the first two phases, partners involved in WP1 and WP2 have provided a general understanding and integrated basis for a cost effective sensors development. Within the following WPs 4 to 8 the new sensors were created and integrated into different identified platforms. During the third phase of field testing (WP9), partners have deployed precompetitive prototypes at chosen platforms (e.g. research vessels, oil platforms, buoys and submerged moorings, ocean racing yachts, drifting buoys). Starting from August 2015 (month 22; task 9.2), these platforms have allowed the partnership to test the adaptability and performance of the in-situ sensors and verify if the transmission of data is properly made, correcting deviations.

In task 9.1 all stakeholders identified in WP2 have been contacted in order to agree upon a coordinated agenda for the field testing phase for each of the platforms. Field testing procedures (WP2) and deployment specificities, defined during sensor development in WPs 4 to 8, have been closely studied by all stakeholders involved in field testing activities in order for everyone to know their role, how to proceed and to provide themselves with the necessary material and equipment (e.g. transport of instruments). All this information have provided the basis for designing and coordinating field testing activities.

Subsequently, the available new sensors have been tested since August 2015 till mid-October of the current year (2016) as part of task 9.2, following the indications defined in D9.1, such as the intercomparison of the new sensors with commercial ones, when possible.
The availability of new sensors was quite different in time starting with the first tests in September and October 2015 on noise, nutrient and heavy metals sensors and closing with pCO_2 in late September 2016.
Sensors are technically fully described in the deliverables of WPs 3 to 8 and are here just mentioned where necessary. For further details, please consider those reports.

Objectives and rationale

The protocols prepared in D9.1 have been verified during the field testing activities of the innovative sensors on platforms. These can be summarized into 3 categories: (1) Research vessels (regular cruises); (2) Fixed platforms; (3) Ocean racing yachts. An exhaustive analysis of the different data obtained during field testing activities has been carried on in order to set possible optimization actions for prototypes design and performances. The data from each platform have been analyzed to verify limits and optimal installations or possible improvements. Finally a set of possible optimization actions has been defined. Data and observations collected during the course of field testing have been used to iteratively optimize the design and performance of the precompetitive prototypes.

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1 INTRODUCTION

1.1 Background

In Task 9.1 ("Design, coordination and implementation of the field testing activity") the aim was to start (at month 18) a coordinated agenda for the field testing phase for each platform used for testing the new sensors realized in the framework of the COMMON SENSE project. Field testing procedures (WP2) and deployment specificities defined during sensor development (WPs 4 to 8) have been closely studied by all people involved in field testing activities in order for everyone to know their role, how to proceed and to provide themselves with the necessary material and equipment (e.g. transport of instruments). All these have provided the basis for designing and coordinating field testing activities realized in Task 9.2 ("Field testing, Validation and Optimization report") and descried in the following chapters of this report.

Type and characteristics of the system (vessel or mooring, surface or deep, open sea or coastal area, duration, etc.) used for the field testing activities have been planned above-mentioned descriptors related to eutrophication, concentration of contaminants, marine litter and underwater noise. Developers limited the use of their sensors to the surface (first 5-10 meters depth) or outside the water through the analysis of water samples. This has reduced the type of platforms to be used; so several platforms, like deep moorings, available and described in D9.1 were not taken in consideration.

1.2 Organisation of this report

This report provides information on the testing activities, and subsequent results, realized on sensors for eutrophication, microplastics, heavy metals, underwater noise, plus additional new sensors for innovative piro- and piezo-resistive polymeric temperature and pressure, nanosensors for autonomous pH and pCO_2 measurements.

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2 METHODOLOGY

2.1 Sensors synthetic description

As mentioned in the previous chapter and shown below, at the present stage most of the sensors could only be used in the upper 5-10 meters and needed of frequent maintenance (see Annex 1).

Innovative pyro- & piezo-resistive polymeric temperature sensors (WP4: resp. CSIC): measurements are to be directly performed by immersion into the water with max 100 m depth with no maintenance as the sensors are located within a small container and the material is stable for years, but requires periodical calibration.

Voltammetric and Resistivity nanosensors for autonomous pH and pCO_2 (WP4: resp. CSIC. Resp. for sensor development: FTM-UCIM, CNR-IPCB and DROPSENS**):** deployment in surface waters (0-5 m). Sensors are disposable and intended to be used for a limited number of cycles; maintenance interval will depend on sampling frequency. Distilled water and two buffer solutions may be necessary for periodic reconditioning of the electrodes.

Eutrophication (WP5: resp. DCU): usable in surface waters (0-3 m depth) with targeted maintenance interval of 1 month (if storage capacity of reagent, calibration and waste storage containers is sufficient). Sensor operates using battery power.

Microplastics (WP6: resp. LEITAT): operationally automated to minimize human activities; sensor could be placed directly in water (so that a sampling system is not needed); additional discrete sensors are included in the sampling system like turbidity, fluorescence, CTD, pH, DO_2 with a sampling frequency will be set at 30 minutes. Can be deployed at depths of up to 100 m.

Heavy metals (WP7: resp. DROPSENS): usable in surface waters (0-5 m) with no maintenance as it is single use, but only the fluidic system might need maintenance against fouling. The sensor, fully automated, incorporates several containers: A) two liquid reservoirs with two types of buffer solutions; B) three containers with standard solutions of different concentrations for each the heavy metals under study; C) an additional container to collect the residual liquids containing heavy metals.

Underwater Noise (WP8: resp. CEFAS): to be used only near the surface (0-5 m) and deployed/installed using low noise platforms (e.g. fixed quiet *surface moorings* or maybe also on *surface drifting buoys*);

Sensors for	Sensors platforms
1. Innovative piro- and piezo-resistive polymeric temperature sensor (WP4) 2. Nanosensors for autonomous pH and pCO_2 measurements (WP4) 3. Eutrophication (nutrients; WP5) 4. Microplastics (WP6) 5. Heavy metals (WP7) 6. Underwater noise (WP8)	The available research platforms: (A) Research vessels (regular cruises); (B) Oil platforms; (C) Buoys and submerged moorings; (D) Ocean racing yachts; (E) Drifting buoys.

Table 1. Sensors to be deployed and available platforms

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2.2 Available platforms

The research platforms available for the field testing (by partners indicated in brackets) were the following (see also in Annex 2 where some available platforms are described more extensively):

A. Research vessels

MINERVA UNO (resp. CNR): substitutes R/V URANIA; all sensors could be tested onboard during 15-days long cruises in 2015 and 2016, mounted on frame of CTD/rosette system or downflow of an on-board seawater pump. Sensor for *microplastics* could be tested on nets but with autonomous power (daily sensor maintenance when on board).

OCEANIA (resp. IOPAN): all sensors could be tested. Five winches were available, two with cable line. There were no strong restriction in number of instruments/winches operating simultaneously, unless sounding depth was higher than 50 m. Once onboard there was the possibility of constant maintenance of mounted sensors.

Motorboat (resp. IOPAN): all sensors could be tested with limited dimensions and weight of the embarked instruments. One winch was available, with a sounding depth of up to 50 m. Once onboard there was the possibility of constant maintenance of mounted sensors. Work performed up to 2 B, wind up to 6 m/s, wave up to 1 m with the Gulf of Gdańsk area and Vistula river as areas of operations.

SARMIENTO DE GAMBOA – SdG (resp. CSIC): all sensors could be tested onboard during several cruises planned yearly. Testing of sensors for temperature, pH, pCO2 and nutrients was offered in a July 2016 cruise where observations of those parameters were planned although embarkation of developers was not possible as fully booked by the cruise responsible embarked for other deployments.

B. Oil platforms

In Gdansk Bay, Southern Baltic (resp. IOPAN): sensors limitations in size and weight (access subject to agreement with the platform administration), and in number due to security reasons. The maintenance was possible monthly. Restrictions and late availability in the project lifetime strongly limited its use.

The Casablanca platform in front of Barcelona (resp. CSIC): sensors for eutrophication, innovative piro- and piezo-resistive polymeric temperature sensor, nanosensors for autonomous pH and pCO_2 were installed underwater or to a pump if not submersible, using batteries or power supplied by the platform. Unfortunately, due to new security regulations, green light for its use arrived too late for the project lifetime.

Smartbuoy (resp. CEFAS): all sensors were available for testing, but size of sensors/instruments was a constraint for prototype units. Therefore only the noise sensor has been tested here and was mounted in such a way to avoid pickup from the mooring.

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C. Buoys and submerged moorings

Oceanographic submerged moorings in the Mediterranean (resp. CNR): three deep mooring lines available (two in the Sicily Strait and one in the Corsica Channel). Only deep sensors were tested and their number depends on their dimension and weight due to strong currents and also on depth pressure limit (possibly at different depths ranging 150-400m). Maintenance was planned every six months. Due to these limitations none of the sensors has been tested in this platform.

Deep moorings at the continental slope and canyons of the NW Mediterranean (resp. ICM-CSIC): None of the sensors has been tested here since as in the previous platform, only deep sensors long time lasting, provided enough power in batteries, could be mounted considering that moorings maintenance was planned about once a year.

Aqualog undulating mooring (resp. CSIC): The proposition was to install the same kind of sensors as in the previous listed moorings taking in consideration its monthly maintenance. No adequate sensors were available for installation in this platform.

OBSEA Underwater observatory (resp. CSIC): with the same availability like Aqualog. It is part of the Fixed point Open Ocean Observatory network (FixO3; http://www.fixo3.eu) that offered also the other sites to test sensors. Request to submit in annual transnational access (TNA)calls.

Fixed buoy in front of Barcelona (resp. CSIC): At the end of 2016 a fixed surface buoy, with data radio direct link with the ICM, will be installed in front of the Barcelona yacht harbour. Temperature sensor is planned to be installed here. Other autonomous light sensors can also be installed on demand (see drifting buoys described below).

D. Ocean racing yachts

IMOCA Open boats (resp. FNOB): available for sensors of microplastics and eutrophication. As racing boats, installation depended on sensors size and needs. They could be mounted into the systems provided that fit inside the boat (for example water treatment or water treatment engine cooling circuit). Platform was relocatable depending on needs and viability.

E. Drifting buoys.

Drifting buoys (resp. ICM-CSIC): theoretically sensors for eutrophication, microplastics, innovative piro- and piezo-resistive polymeric temperature and pressure sensors, could be mounted even if size and weight of sensors as sensors battery power capacity strongly limited their use.

Further platforms

During the project several other platforms have been proposed and some of them have been used like the Arctic base "Dirigibile Italia" in Svalbard archipelago proposed by CNR, the Lowestoft harbour in UK proposed and used by Cefas and several stakeholders interested in evaluating the sensors. A list follows:

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The CNR Arctic base *Dirigibile Italia* (http://www.polarnet.cnr.it) in Ny-Ålesund (78°55' N, 11°56' E; Norway), north-western part of the island of Spitsbergen in the Svalbard archipelago, was available for testing some of the sensors in extreme climatic and meteo-marine conditions in order to check their environmental limits at sea (at local wharf or on a boat).

Dr. Inga Lips from the Marine Systems Institute of the Tallinn University of Technology in Estonia, in agreement with LEITAT, offered to test the microplastics sensors in the Baltic Sea. They had two ferrybox systems (one on board passenger ferry and the other on board their research vessel) and two autonomous profiling buoy stations.

CNR proposed its *Acqua Alta* research tower offshore the Venice Gulf on 16 m of water depth. It consists of a platform with an instrument house, supported by a steel pipe structure, similar to that of an oil well derrick. Given the high level of security on board and wide desk space, sophisticated instruments could be hosted on board reducing drastically the risk of loss. It is part of the Long Term Ecological Research network http://www.ve.ismar.cnr.it/piattaforma/

DCU obtained the availability of a Floating Pontoon by the Hawai'i Institute of Marine Biology (HIMB; http://www.hawaii.edu/). It consists of a floating pontoon in a small harbour managed by HIMB where institute's boats and small vessels are docked. Depth is just a few meters but the area is controlled and there is the availability of electricity.

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3 RESULTS AND DISCUSSION

3.1 Introduction

The activity started at month 22 (August 2015). Tables with the list of all external testing activities scheduled and realised have been created (named TESTING ACTIVITIES.XLSX, resumed in Annex 3) and that partners have continuously updated and made available on the account on Basecamp for the project under WP9. Table 2 summarizes the testing activities realized before September 2016 thanks to the strong collaboration between partners.

Sensor	Platform	Location	Date	Participants
Temperature	Laboratory setup	Barcelona	02/07 + 22/10/15	CSIC
Temperature	Oceania vessel (IOPAN)	Gdańsk (PL)-Tromsø (N)	13-20/06/16	CSIC, IOPAN
pH (Res.)	Laboratory	FTM - Skopje in simulated SW	27-28/10/15+ 29/03/16+20-27/04/16	FTM-UCIM
pH (Res.)	Laboratory	on samples from Oristano lagoons	21 -22/04/16	FTM-UCIM
pCO_2 (Res.)	Laboratory	on samples from Lucrino (Naples Gulf; Italy)	10/06-15/06/16	FTM-UCIM
pH (Volt.)	Oristano lagoons	Oristano area (I)	21-22/03/16	CNR, DROPSENS
pH (Volt.)	Oristano lagoons	Oristano area (I)	21-22/04/16	CNR, DROPSENS
pH (Volt.)	CNR Arctic base	Svalbard Archipelago (N)	19/06-04/07/16	CNR, DROPSENS
Eutrophication	Floating Pontoon	Hawai'i Institute of Marine Biology (USA)	13/09-23/09/15	DCU, TelLaB
Eutrophication	Minerva Uno vessel	Western Medit. Sea	25/11–14/12/15	DCU, CNR
Eutrophication	CNR Arctic base	Svalbard Archipelago (N)	19/06-04/07/16	DCU, CNR
Microplastics	RV Minerva Uno	Western Medit. Sea	25/11–14/12/15	IDRONAUT, CNR
Microplastics	King fisher yacht	Barcelona (E)	April 16	FNOB, LEITAT
Microplastics: Analyzer	Oceania cruise	Gdansk (PL)-Tromsø (N)	13-20/06/16	IOPAN, LEITAT
Microplastics: MISS System* & Analyzer	CNR lab &I Mistras lagoon (Oristano)	Oristano area (I)	26-28/09/16	IDRONAUT, LEITAT, CNR
Microplastics: Analyzer	Vendeé Globe	Worldwide	November 16	FNOB, LEITAT
Heavy metals	Minerva Uno vessel	Western Medit. Sea	25/11–14/12/15	DCU, CNR, DROPSENS
Heavy metals	CNR Arctic base	Svalbard Archip. (N)	19/06-04/07/16	DCU, CNR, DROPSENS
Underwater noise	Local harbour	Lowestoft (UK)	02-04/09/15	CEFAS
Underwater noise	Motorboat	Gdańsk Bay (PL)	29/10–10/11/15	CEFAS , IOPAN
Underwater Noise	Oceania cruise	Gdansk Bay (PL)	6/11 – 7/11/15	IOPAN

Table 2. List of testing activities realized in the framework of WP9

* Microplastics Integrated Sampling System

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- **Temperature, pressure, pH, pCO_2 - WP4 (resp. CSIC)**

Innovative pyro- & piezo-resistive polymeric temperature and pressure sensors (CSIC)

I testing – CSIC 2nd July and 22 October 2015: laboratory temperature controlled tests in sea water in Barcelona under a wide temperature ranges.

II testing – CSIC/IOPAN 13th – 20th June 2016: temperature tested on Oceania cruise from Gdańsk (Poland) to Tromsø (Norway)

Pressure sensor is not listed here because tested in laboratory.

Voltammetric nanosensors for autonomous pH sensor (FTM-UCIM, CNR, DROPSENS)

I testing - DROPSENS/CNR. 21st - 22nd March 2016: the two pH sensors realized by Dropsens and CNR were tested using water samples collected in the Oristano Gulf and compared with pH sensors from Sassari University (stakeholder).

II testing - DROPSENS/CNR. 21st - 22nd April 2016: the two pH sensors realized by Dropsens and CNR were tested using water samples collected in the Oristano Gulf and compared with pH sensors from Sassari University (stakeholder).

III testing - DROPSENS/CNR. 19th June- 04th July 2016: at the CNR Arctic base in Ny-Alesund (Svalbard Archipelago, Norway) pH measurements were done on water samples collected in the local fjord during 4 daily cruises.

Resistivity nanosensors for autonomous pH & pCO_2 (FTM-UCM, CNR)

I testing - FTM-UCIM/CNR 27th-28th October 2015: tested pH in artificial sea water at FTM laboratories

II testing - CNR 21st - 22nd April 2016: water sampled in Oristano and analyzed for pH at CNR laboratories in Naples

III testing - FTM-UCIM/CNR 29th March and 20th-27th April 2016: tested pH in artificial sea water at FTM laboratories

IV testing - FTM-UCIM/CNR 10th-16th June 2016: tested pCO_2 in sea water in Lucrino Gulf, Naples (I)

Eutrophication - WP5 (resp. DCU)

I testing – DCU/TelLab 13th – 23rd September 2015: Floating Pontoon at Hawai'i Institute of Marine Biology (Hawaii, USA). The following components have been tested altogether: linear dosing pumps, epi-pump peristaltic pump, diaphragm pump, ccommercial temperature, pressure and humidity sensor.

II testing – DCU/CNR 25th November – 14th December 2015: CNR cruise Ichnussa2015 (R/V MINERVA UNO) in the Western Mediterranean with the acquisition of hundreds of samples compared with the analyses from the laboratory from ENEA UTMAR in La Spezia (Italy).

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III testing - DCU/CNR 19th June- 04th July 2016: CNR Arctic base in Ny-Alesund (Svalbard Archipelago, Norway). Over 100 water samples have been collected in the local fjord during 4 daily cruises and the analyzed in the laboratory at CNR base.

- **Microplastics - WP6 (resp. LEITAT)**

I testing - IDRONAUT/CNR 25th November – 14th December 2015: CNR cruise Ichnussa2015 (R/V MINERVA UNO) in the Western Mediterranean. IDRONAUT provided information about the installation and field requirements of the MISS microplastics analyser sampling system. Moreover, IDRONAUT prepared a short guide describing how to prepare and configure the Microplastics Integrated Sampling System (MISS system) before deployment and instructed the CNR partner on these procedures. Practice in the laboratory was carried out in November 2015 before the cruise.

II testing - LEITAT/FNOB May 14th 2016: LEITAT performed a pre-deployment, prior to the testing scheduled for May 15th, on board the King fisher to test the power source and the water flow and Pump on the boat. LEITAT, FNOB and King Fishers' technical team worked together to find the best solution as far as the power consumption and connections and to find the best place to install the sensor on the boat. On 14th May, after improvement done on microplastics sensor, it was tested on Mediterranean sea for half-day. Coastal navigation close to Barcelona.

III testing – LEITAT/IOPAN 13th – 20th June 2016: Microplastics sensor was installed, deployed and tested on Oceania cruise from Gdańsk (Poland) to Tromsø (Norway)

IV testing – IDRONAUT/LEITAT/CNR 26th – 28th September 2016: Analyzer and MISS System were deployed on CNR facilities and in the Mistras lagoon close to ORISTANO (I). Integration with SSU and MISS was tested and validated The MISS deployment allows to acquire on a time basis (5 minute) reference physical, chemical and optical data about master sea-water parameters, from a traditional CTD installed on the MISS system and to collect five (5) 0.8 liter samples for comparative laboratory analysis.

V testing – LEITAT/FNOB 6th November 2016: Microplastics sensor was installed on IMOCA racing yacht participating at the Vendeé Globe event. At the end of the race, data will be recovered and further analyzed (after the submission of this report).

- **Heavy metals - WP7 (resp. DROPSENS)**

I testing - DCU/DROPSENS/CNR 25th November – 14th December 2015: CNR cruise Ichnussa2015 (R/V MINERVA UNO) in the Western Mediterranean with the acquisition of samples to be run through the heavy metals sensor system for performance testing.

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II testing - DCU/CNR 19th June- 04th July 2016: CNR Arctic base in Ny-Alesund (Svalbard Archipelago, Norway). Over 50 water samples have been collected in the local fjord during 4 daily cruises and the analyzed in the laboratory at DCU in Ireland.

- **Underwater noise - WP8 (resp. CEFAS)**

I testing - CEFAS 2nd-4th September: Lowestoft harbour (UK)
Lowestoft harbour, adjacent to the Cefas quay (52° 28' 23.6"N, 001° 44' 38.56"E), provided us with good security, access to the water's edge where we could use a crane to lower and raise the system to the water. A record of passing ships was obtained from the harbour master, so that we could correlate to noise events logged by the system. Although not an ideal site due to shallow water depth (approx. 6m tidal) and in close proximity to the harbour wall, the site enabled us to gather useful performance data. This first successful deployment of the prototype noise sensor has been completed by CEFAS and we are reviewing the collected data (as part of the D8.3 Report with recommendations of further improvements to follow during the second test).

II testing - CEFAS/IOPAN 29th October – 10th November 2015: cruise in the Gdansk Bay
The setup was deployed on the sea bottom for about 24 hours, and later for a further 12 hours. The equipment worked perfectly. Due to 24 bit A/C converter the dynamic of recording covered the broad range of noise from ambient noise characteristic of sea state 1 and nearby passing ships.
A freely floating sphere was attached to the frame, for security reasons. This resulted in a characteristic ringing (disturbance) to be observed in the time series data, which would not be present in longer term deployments on a custom buoy with quiet mooring.

III testing – IOPAN 13th July, 19th July, 21 July 2016: motor boat in Gdansk Bay
The final production housed prototype was deployed from a bespoke buoy. The logging electronics performed correctly, being triggered to log data every 10 minutes by an off the shelf GPS system. Unfortunately, this field trial proved inconclusive as the data appeared to be intermittent. Initially we believed the hydrophone sustained damage when it was dropped. However, further testing indicates the cause to be saturation of the amplifier stage, caused by higher levels of ship noise than was anticipated, and resulting from the increased gain at the hydrophone. The internal gain could not be adjusted at sea, as it was deployed from a small boat. There was no method of sending the summary data back for analysis, as it was triggered by a standard GPS unit not the Master Logger. That said, it can be seen that the use of the highest gain hydrophone, reduces the need for later gain stages, improving the noise figure of the system.

IV testing – Oristano 26th September – 28th September: IAMC moored small research boat.
This test focussed on the full integration with the SubCtech master logger. The sensor correctly logged data upon request from the SubCtech master logger, sending the summary data for onward transmission to the web platform.

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3.2 Temperature, pressure, pH, pCO_2 - WP4 (CSIC)

3.2.1 Protocols history of sensor/s;

Pressure

Two different prototypes of pressure sensors based on a polymeric membrane previously developed at Nanomol-ICMAB-CSIC were prepared with the same kind of connections as those used for the temperature sensor. Both prototypes were then tested in the laboratory using air pressure controlled by a gas manometer (see D4.4 for further information). After realizing that sensors clearly responded to pressure changes, we assumed that further development for sea testing of these sensors would involve (i) housing, (ii) a new PCB adapted to the sensor output range and (iii) external connections with the SSU. Since these steps were the same (or equivalent) as those corresponding to the temperature sensor, it was decided to concentrate all the efforts to this last sensor whose results could be used almost straightforward for pressure sensor tests. Thus, up to now, only temperature sensor has been tested at sea.

Temperature

Four different prototypes of temperature sensors have been developed, all of them based on the same sensing material, an organic bi-layer film of organic crystalline conductors over polycarbonate polymer. The temperature sensitivity of the new material is one order of magnitude higher than the one of Pt_{1000} and shows a linear response, very good performance and stability in laboratory tests (see D4.4 for further information). The material has been developed at the beginning of the project at Nanomol group of ICMAB-CSIC. For all prototypes, first tests have been performed at laboratory level.

First prototype. The sensing material with contacts have been mounted into a plastic container together with a standard temperature sensor Pt_{1000} for temperature control. Sensitivity of the new material is one order of magnitude higher than the one of Pt. According to the tests in sea water at ICM-CSIC laboratory the container was not appropriate for sensing in sea water because it contained too much air, causing a too slow response to temperature, as compared to an external Pt_{100} control sensor.

Second prototype. The container was a stainless tube 5 mm in diameter and 30 cm in length. The sensitive element was placed inside of this tube as close as possible to one of its ends and a connector at the other end. The tests results in sea water at ICM-CSIC laboratory were very good in terms of sensitivity, linearity, reproducibility and reversibility. The sensor was also stable in time. With this prototype the use of the PCB prepared in Tyndall UCC to produce an analogue voltage has also been tested with good results.

Third prototype. Three sensors have been developed. The container was a very thin brass cover of around 1 cm^2 in area and connected to the above mentioned PCBs. Tests in laboratory were OK.

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Tests in Oceania cruise using an electronic board to be connected to a computer through an USB port, developed at the ICM-CSIC, were performed with this prototype. The results indicate a good sensitivity response as compared with a CTD but contacts, housing and sealing against water leaking should be improved (see Annex 4).

Fourth prototype. New robust container for the sensing film made on marine brass, and developed at the ICM-CSIC has been used. The connections and cables were protected inside an adapted Teflon tube (figure below). Tests in laboratory were OK. Tests in Oristano using the PCB and the USB electronic board above mentioned indicate that the whole system has shown good sensitivity and linear response, quite good accuracy but an uncontrolled shift had been observed whose origin is being carefully studied in the laboratory.

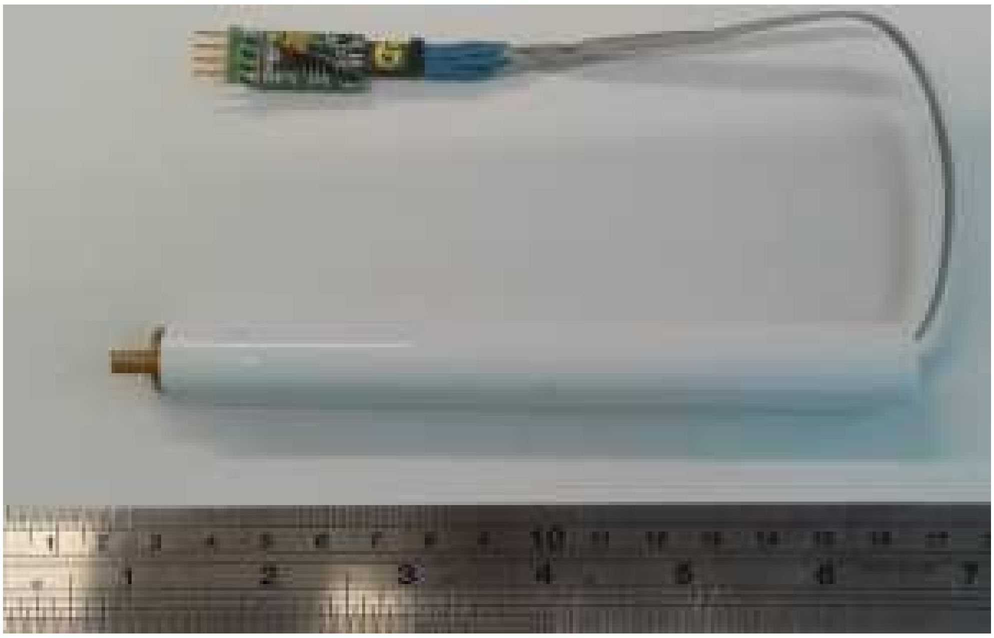

Fig. 1. New housing for temperature sensor attached to the Tyndall PCB

Nanosensors for autonomous pH and pCO_2 measurements

pH sensing with nanocomposites developed at FTM-UCIM:

A first sensor based on G/PANI and MWCNT/PANI nanocomposite resistivity changes was designed and developed as follows:

In the 1st stage G/PANI and MWCNT/PANI nanocomposite powder was obtained by electropolymerization and completely characterized.

In the 2nd stage, nanocomposite tablets were obtained and characterized for resistivity changes as a function of pH. A 4-Probe method was used to confirm the resistivity changes as a function of pH variation in simulated media. Laboratory testing of the G/PANI and MWCNT/PANI nanocomposite tablets by the 4-Probe method was done in Tyndall national Institute, University College Cork.

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In the 3rd stage, G/PANI and MWCNT/PANI nanocomposite sensing part were directly electropolymerized on the SPE gold wires.
The **first nanosensor electrodes** were delivered on the **24M** meeting in Barcelona by FTM-UCIM.

The obtained SPE electrodes were connected with universal multimeter and inserted in the water sample. In this method, the pH measurements were performed by determination of the resistivity [Ω] changes between electrodes due to the changes of H^+ concentration (as a function of pH variation). This difference was correlated to the pH changes of test solutions. In parallel, also the temperature was followed.

The validation of the sensor has been performed in the laboratories of the FTM-UCIM (Macedonia) and CNR (Oristano, Italy). The results obtained showed that the calibration of the pH vs R [Ω] linearly decreased by increasing the pH over the range 7 to 9.2 .

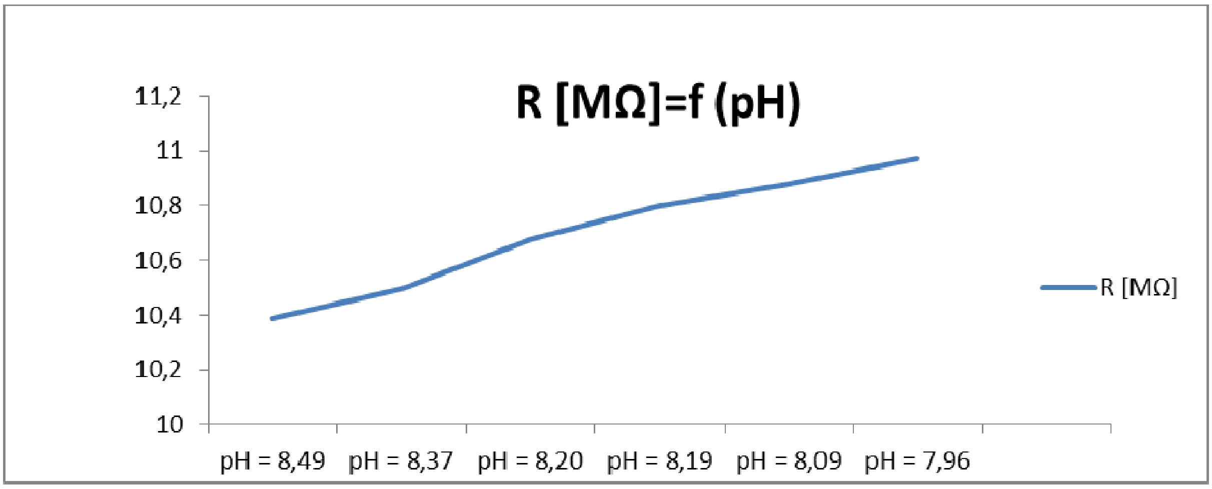

Fig. 2. Validation curve for pH range = 7.9 ÷8.5

The following procedure has been established:

1. Connect the electrode with the cable and introduce the electrode in the water sample
2. Press the start/stop button and keep the electrode inserted in the sample during 120 s.
3. Record the 10 values showed in the display, up to the stabilization of the value (about 6-8 min).
4. Validation of the sensor has been performed correlating the mean of the 3 resistivity measurements with the pH measured with a standard laboratory pH-meter.
5. Wash the electrode thoroughly with deionized water and let it dry before to perform the next measure.
6. The results show that the produced electrodes exhibited good stability within a stabilization time of 12 min. It was also found that recovery time was fast, shorter than 1 min.

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Sensors based on similar materials but with an alternative measurement approach, cyclic voltammetry, were developed and tested for pH sensing. This measurement setup is similar in design to other sensors developed in COMMON SENSE project, which can be convenient for integration.

The devices are based on Carbon Screen Printed Electrodes in which the working electrode (WE) is modified with polyaniline (PANI), an intrinsically conductive polymer, containing Multi-wall Carbon Nanotubes, conductive materials with high aspect ratio and low percolation threshold. The selection of such components is due to the sensitivity of PANI redox behaviour to pH, already reported particularly in acidic range, and to the MWCNTs ability to enhance the electric properties of the materials in which they are embedded. The preparation of the active sensors is based on the simple and fast drop by drop deposition on the WE of the mixture of PANI and MWCNT in organic solvent, then dried in oven.

The best composition of the nanocomposite material (3 wt% of MWCNTs, concentration of PANI solution 1 wt%) to be deposed has been selected testing different systems, containing 1, 3, 5 and 10 wt% of nanotubes. Also the drop by drop deposition method has been optimized by adjusting the number of drops delivered onto the working electrode.

The devices require an immersion in the water samples of 30 sec, then in 1 minute a voltammogram is recorded whose significant data is provided by the anodic peak at positive current values. The potential usually applied for the measurement is in the range -0.8 / +0.8 V, performed with a scan rate of 0.05 V/s.

The range of pH between 7 and 9, interesting for sea water pH sensing, has been particularly investigated with the PANI-MWCNT sensors that displayed satisfactorily linear response.

Determination of pH using DropSens antimony screen printed electrodes:

A second type of sensor developed and tested for pH sensing is based on antimony. Specifically, the sensor used is a screen-printed electrode containing an antimony working electrode and a Ag/AgCl reference electrode, where the antimony acts as a metal – metal oxide system for the determination of pH.

The source of pH sensitivity of metal- metal oxide electrodes is the redox equilibrium between these two phases. As an exception, the antimony surface is spontaneously covered by an oxide layer in air atmosphere, is for that reason that this type of metal has been chosen. The electrode is connected to a small, hand held single-technique potenstiostat, containing a display.

Measurements of pH are performed using zero current galvanostatic potentiometry (ZCP). In this technique, the pH measurement is done by determination of the difference of potential between reference and working electrodes. This difference of potential is correlated to pH changes of test solutions. Reference and counter electrodes are short-circuit in order to have a two electrodes system.

The validation of the sensor has been performed in the laboratory of DropSens (Spain). The results obtained showed that the calibration of the pH vs E (V) remains linear over the range 2 – 10.9 when using Britton- Robbinson 0.1M solutions at 23°C ($r^2 > 0.99$). The reproducibility between same pH values is ≤ 4% with a resolution of 0.2 pH units.

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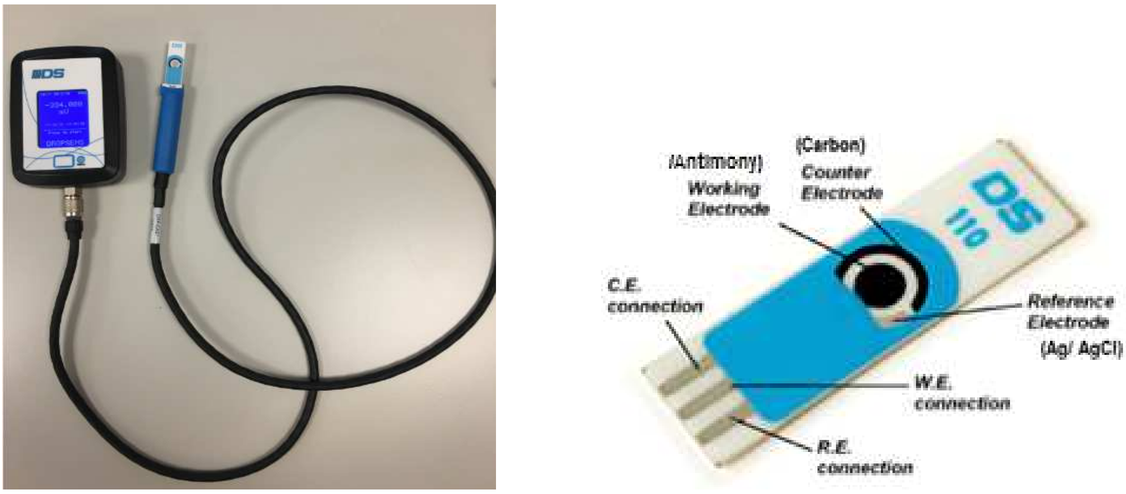

Fig. 3. (a) Potentiostat, connector and screen printed electrode; (b) Antimony screen printed electrode

Once the sensor has been optimized, the electrochemical device (potentiostat) has been configured with the ZCP technique and its specific parameters. To facilitate the reading of the measurements, after the measurement takes place, the results are displayed in a LCD screen. Moreover, displayed results are recorded internally and can be downloaded via USB to a PC. During field testing activities, it has been decided to record the results manually after each measurement. In addition, to be able to compare results between different operators, it has been established the following procedure for the measurement of the samples:

1. Introduce the electrode in the sample
2. Press the start/stop button and keep the electrode inserted in the sample during 120s.
3. After time finished (120 s) press the start/ stop button again
4. Record the 3 values showed in the display, corresponding to the last 3 measurements.
5. Validation of the sensor has been performed correlating the mean of the 3 potential measurements with the pH measured with a standard laboratory pH-meter.
6. Wash the electrode thoroughly with deionized water and let it dry before to perform the next measure

3.2.2 *Exhaustive analysis of the data collected during field testing activities including inter-comparisons with commercial sensors;*

Temperature

<u>I testing:</u>

Two sessions of testing in seawater (02/07/2015 and 22/10/2015) have been performed in the ICM-CSIC laboratory with the second prototype sensor. A set-up especially designed for such testings (see D4.4 for further information on these tests) enabling different temperatures within a wide range (3 to 33°C). Electronic used for measurements was an Agilent 34970A data acquisition/switch unit from ICMAB-CSIC laboratory connected to a Pt_{100} high precision temperature probe.

The main results of these first testing sessions were:

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- The sensitivity is better than 0.01 °C
- The sensor has a linear, reproducible and reversible electrical output stable in time
- The power consumption of the sensor varies from 1 to 5 µW

II testing:

It was carried out underway along the S/Y Oceania transit from Gdańsk (PL) to Tromsø (NO) (see the cruise report in Annex 4). Sea surface temperature was continuously measured using the new sensor assemblage and a CTD both receiving sea water pumped to the ship's deck and laboratory. Sensor assemblage consisted on the sensing film protected with a very thin brass cover and connected to a PCB developed by Tyndall UCC to produce an analog voltage. Since SubCtech SSU was still not available, an electronic board to be connected through an USB port was adapted to be used for this testing activity. (see Annex 4 for details).

The main results of this second testing were:

- All the process to prepare the sensor has to be clearly improved. Especially contacts, housing and sealing against water leaking.
- All the ensemble is clearly too fragile against stresses on connexions and cables. It must be improved.
- Sensor resistance ranges should always be kept between two fixed reference points. 10000 and 30000 Ω for the typical seawater temperatures (-2 to 32°C) are quite adequate.
- PCB conditioner was working correctly but if modified for 5V instead of 12V DC power supply, consumption must likely be reduced.
- A/D and processor. All the present experience has been done using a board adapted from another use. It has been working properly but it is not the one developed within the COMMON SENSE consortium.
- The whole system has shown good linear response, quite good accuracy but some uncontrolled shifts had been observed whose origin must be carefully studied in the laboratory.

pH, pCO_2

Results obtained with *pH nanocomposite electrodes developed at FTM-UCIM*

The field testing performed on March 23th 2016 and on September 28th 2016 with, respectively, the water samples from the Oristano lagoons and of the laboratory tank in Torregrande (Oristano) displayed quite good results (more in the following paragraph) if compared with the laboratory glass pH electrode, due to a difference of ±0,3 to ±0,5 points. Even if a quantified sensitivity level of the device has not been determined up to now, anyway the trend in the change of the signal as function of the pH is coherent with the values registered with the commercial instrument.

Using the MWCNT/PANI and G/PANI electrodes (see figure 4), FTM-UCIM has followed the resistivity [Ω] vs. pH of the sea water in the lab of Oristano as well as in the sea water directly in the lagoon of Oristano. In parallel the pH was controlled by conventional pH meter. In the same time, also the temperature of the sea water was measured. The obtained curves are presented in the following two pictures (figures 5 and 6).

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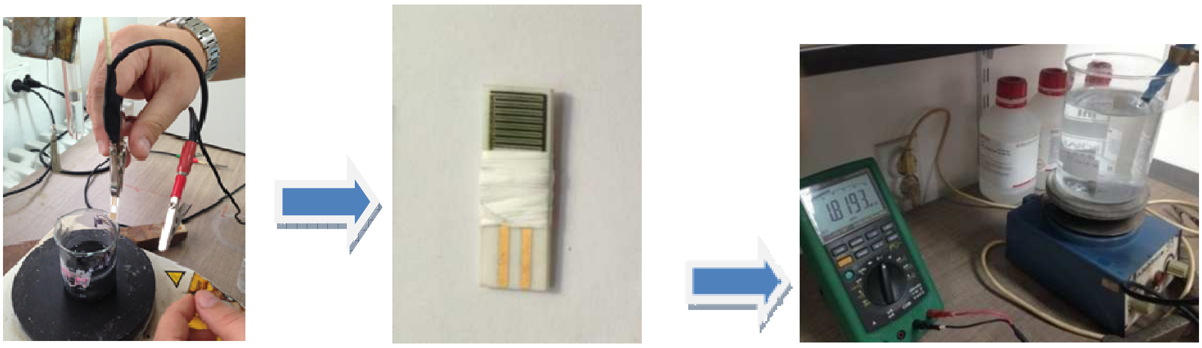


Fig. 4. MWCNT/PANI SPE designed by FTM-UCIM

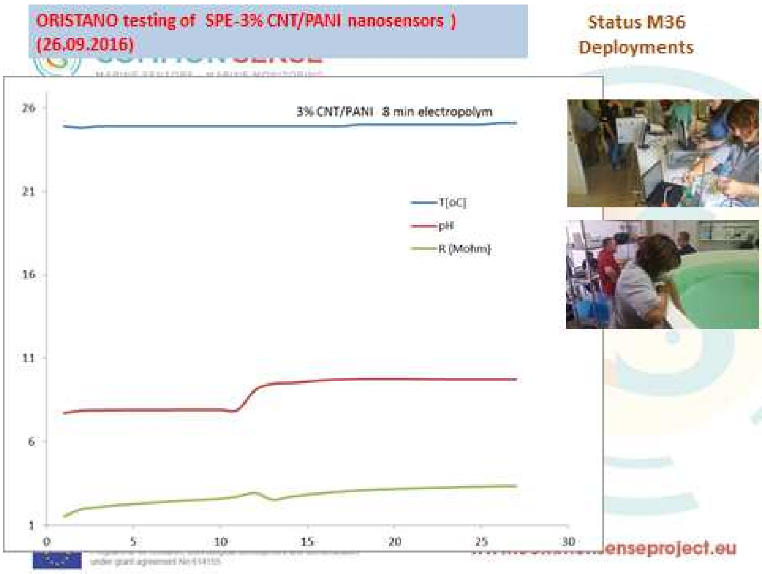
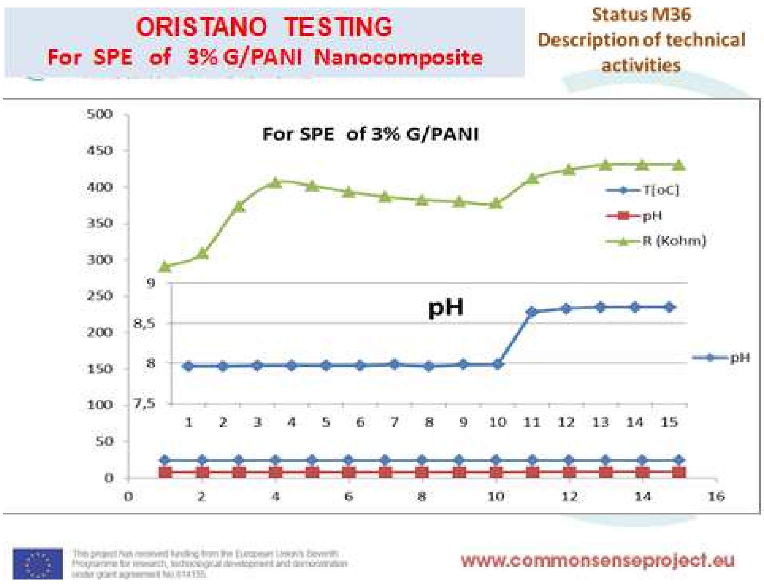


Fig. 5 (above) and 6 (below). Field testing at CNR in Oristano, September 2016 with resistivity [kΩ]on abscissas and time [minutes] in ordinates



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Comparison of the measurements performed in the sample of the lab Tank with the measurements performed directly in the lagoon is presented on the following figure 7. Evidently, the resistivity curves followed the same trend in the similar pH range (around 8).

Namely, presented resistivity curve for 16 minutes at pH=8,1 in the sea water was compared with the resistivity curve for 16 minutes in the lab tank when pH was changed in the range from 7,9 to 8,7 by adding NaOH drops in the waterglass. Evidently, the resistivity curves have similar shape, with the note that the resistivity values are in different range. For constant pH=8,1 they are around 1000 kΩ, while for increasing pH from 7,9 to 8,7, resistivity in the lab tank was measured from 400 kΩ for pH=7,9 to 500 kΩ for pH=8,7.

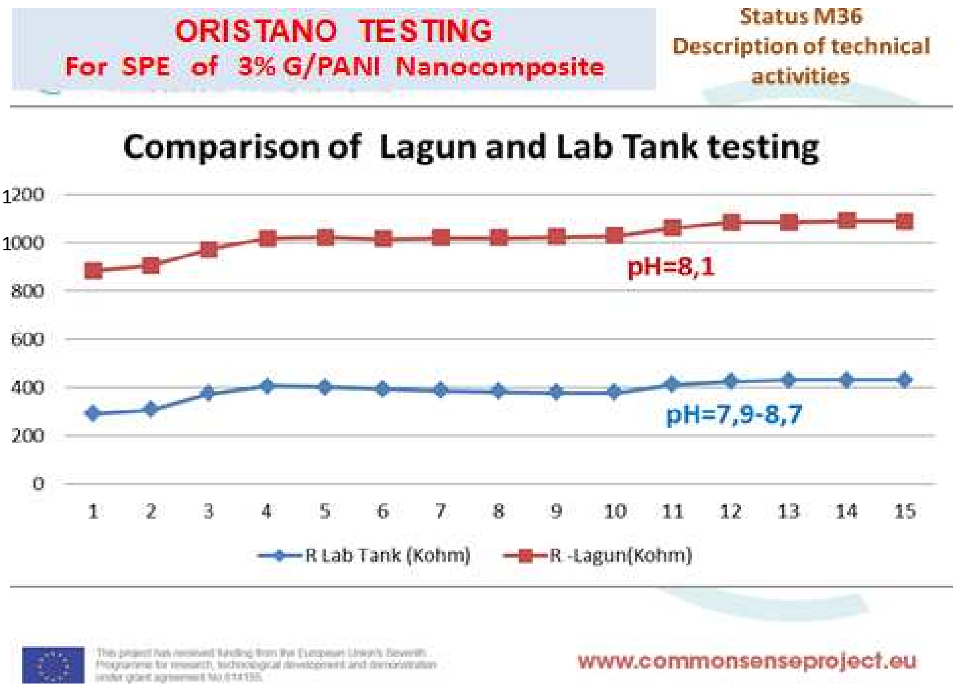

Fig. 7. Comparison of lagoon and Lab-tank testing with resistivity [kΩ]on abscissas and time [minutes] in ordinates

From the first series of tests in Sassari, the need for an improvement of the stability of the signal with the time emerged. Thus, upon the optimization of the devices preparation, in the second series of field tests the registered voltametric signal resulted more stable in time.

Results from voltammetric sensors:

From the first series of test in Sassari (March 2016), the need for an improvement of the stability of the signal with time emerged. Thus, upon the optimization of the devices preparation and an adjustment in voltametric scan parameters, in the second series of field tests the registered voltametric signal resulted more stable in time.

In figure 8, a graph reporting the results obtained by 5 consecutive scans on the same water sample is reported. The reading of the reference pH meter, during the scans, varied between 8.0 and 8.1 (as indicated by the dashed lines.

The agreement with reference pH meter and the repeatability of the measurement are better than 0.1 pH points.

Comparing the results obtained with the same electrode, in runs carried out at few hours one from the other, reproducibility was found to be better than 0.2 pH points.

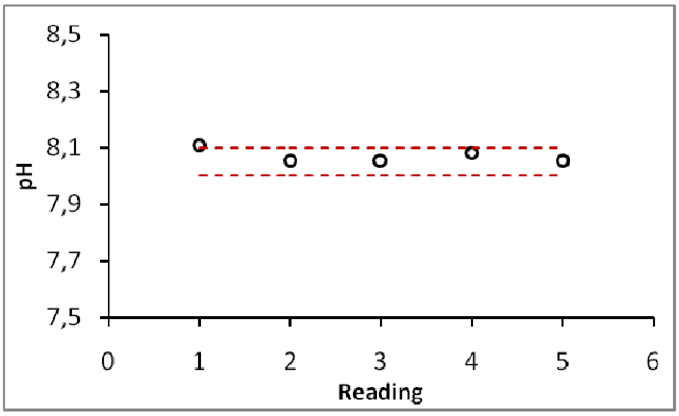

Fig. 8. pH calculated in 5 consecutive scans in sea water (in Oristano) as compared with the range of pH indicated by the reference pH meter (between 8.0 and 8.1).

Results obtained with DropSens pH sensor based on antimony screen printed electrodes

The tests using Dropsens pH sensor, were realized in March and April 2016 with waters sampled in the Oristano lagoon-gulf system. Results were compared with those obtained using stardard pH instruments at at Sassari University and gave some discrepancies in both experiments. New cables have been designed for further tests planned in June in the Arctic, to avoid the entry of water into the connector during the measurement of the samples, as if connections are wet, the data obtained are not reliable.

The average temperature conditions of the Oristano lagoons when samples were collected, during March and April was ~ 20°C, with variable salinity (from 12.28 to 38.50‰). As described before, once the electrode has been inserted in the sample, the potential was recorded after 120 s. The sensor showed reproducibility between potential measurements, with %RSD < 0.6% between different samples with same pH value and with a precision of 0.5 pH units.

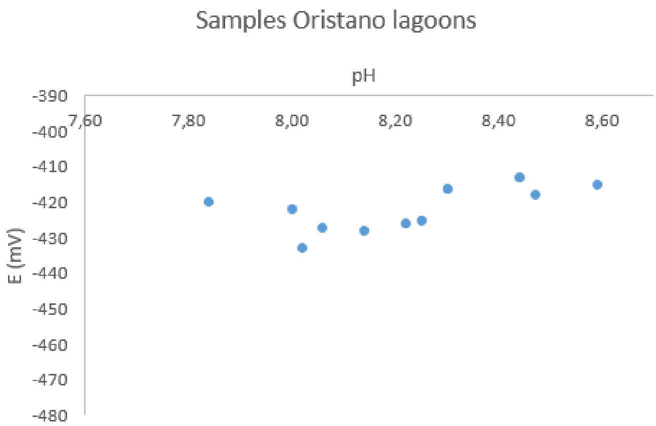

Fig. 9. Mean potential recorded vs pH values: Oristano March-April 2016

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Samples from Arctic base where collected on the 22nd of June 2016. The average temperature of the samples was 3.3°C and salinity varied from 29.2 to 35‰ and depth was lower than 300 m. The sensor showed reproducibility between potential measurements, with %RSD < 1.4 % between different samples with same pH value and with a precision of 0.5 pH units.

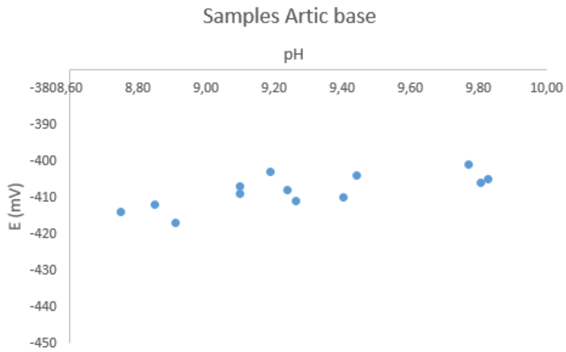

Fig. 10. Mean potential recorded vs pH values: Arctic-June 2016

With all the data obtained during field testing activities it has been concluded that:

- Antimony pH sensor showed a good response in the determination of pH in the range 7.84 – 9.827, in samples with salinity values between 12.28 and 38.5.
- The sensor is temperature sensitive, but results obtained with samples collected at 20 °C and 3.3°C can be compared with good response between different electrodes.
- Precision of the samples has been showed to be 0.5 pH units. This value is not acceptable when measuring seawater samples, so the sensor needs to be optimized.

For more detailed results see the DropSens' pH testing report in Annex 5.

3.2.3 *Short description of stakeholders involved in testing activities with feedbacks;*

Local fishermen were involved during the common tests at the CNR in Oristano at the end of September 2016 with measurements of lagoon water temperature.

For pH and pCO_2. Faculty of Technology and Metallurgy has participated in this field testing, using two nanocomposites systems: G/PANI and MWCNT/PANI systems.

Sassari University was also involved in the testing, providing water samples and comparative pH measurements made with benchtop instruments.

3.2.4 *Possible optimization actions for prototypes design and performances after testing activities;*

<u>Temperature</u>

- Sensor housing has been improved
- Temperature shifts are being carefully studied

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- SSU adaptation is ongoing
- Oscillating response on the SSU has to be addressed

Further points could be added after the end of the analyses of the data acquired during the common test in Oristano, last September 2016. These further points will be part of the final scientific project report in 2017.

pH, pCO_2

The PANI based pH sensors have been tested on a laboratory scale, displaying quite good potentiality. They are suitable for the analysis of samples with pH values in the range required by the target application and they displayed the ability to detect pH in both laboratory (buffers and seawater simulating solutions) and real samples. Actually, it has been verified that the presence of many possible components of a real seawater does not affect the efficacy of the device working, not disturbing the measurements. Even if the response of the instrument to the pH variation adheres quite well to the results of the laboratory pH meters commonly used as reference, it is important to improve the sensitivity of the systems in order to allow more precise results. Thus, it is important to realize a more controlled nanocomposite deposition on the electrode, obtaining a layer with fixed thickness and a constant covering degree of the working electrode surface.

In the end, such devices have to be integrated in a portable system in order to allow the direct use in the field. In particular, a pumping system has to be connected to the device for the seawater to flow and wet in continuous mode the working electrode. A set of calibration curves will be produced for each system. Also, material and production sheets will be designed for each nanocomposite electrode. The precision value obtained during field testing activities showed that the DropSens pH sensor needs to be optimized. The optimization of the sensor includes the use of other electrochemical technique, as potentiometric detection. In this technique, a current is applied during the measurement, so a more stable signal can be obtained. Once the current applied is optimized, the sensor needs to be tested using artificial samples. If a good linear response, reproducibility and precision values are obtained, the sensor can be tested using real sea water samples. The time needed to perform this action has been estimated to be of about 6 - 8 months.

Also, with better resolution of the measurements, it can be possible to have a better precision in the measured potential. This last improvement requires the development of a new instrument which will required more than one year to be ready. The optimization of the sensor will not be achieved in the last months of the COMMON SENSE project, for the reasons explained before.

3.2.5 *Limits and optimal installations or possible improvements and definition of further possible optimization actions.*

Temperature

After issues above are being corrected. sensor will be placed in a fixed buoy to do the last testing within the project. It will remain to test its behaviour on depth under pressure.

pH, pCO_2

Optimal installation is on a boat deck.

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3.3 Eutrophication - WP5 (DCU)

3.3.1 Protocols history of sensor/s;

The COMMON SENSE nutrient sensor is based on a similar approach to the systems previously developed at DCU for autonomous detection of phosphate and other parameters based on a combination of microfluidic analytical systems, colorimetric reagent chemistry, low-cost LED-based optical detection and wireless communications. The work presented in here shows the development and deployment of the pre-competitive prototype for the detection of nutrients in marine environments. Each component was developed, assessed and optimised before being integrated to form a working precompetitive prototype for laboratory and field-testing (Table 3).

Prior to each deployment testing of the precompetitive prototypes was carried out in the laboratory at DCU using reference standards and marine samples sourced by DCU, and Tellabs (figure 11) and is outlined in D5.1 and D5.2 and D7.1 and D7.2. Table 3 shows a summary of the history of the components tested during each deployment. During the initial deployment (field deployment 1) after laboratory validation three different pumping mechanisms, varying tubing and inlet filters were tested in real conditions for a period of ten days to evaluate how each components suitability for incorporation into the deployable system.

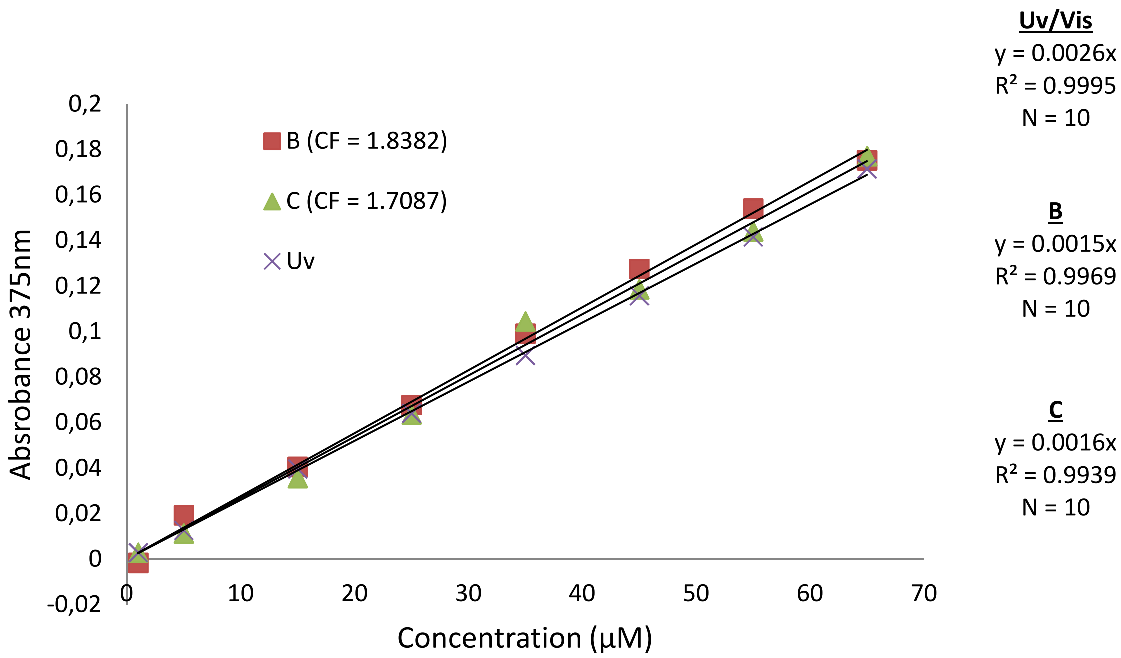

Fig. 11. Inter-laboratory results obtained using Phosphate standards provided by TelLaB (1-65µM). The graph shows the comparison of the UV-Vis benchtop system (UV-Vis) the COMMONSENSE benchtop system B and C

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Field Test No.	Location	Platform	Components Tested during each deployment
1	13th Sept – 23rd Sept 2015: Hawai'i Institute of Marine Biology	Floating Pontoon	• Inlet filter • Inlet pump • Temperature sensor • Light sensor • System firmware • Tubing • Reagent stability
2	25th Nov – 14th Dec 2015: CNR cruise in the Western Mediterranean	R/V Minerva Uno	• System Firmware • System Electronics • LED,PD lifetime and efficiency • Chemistries for each nutrient Reagent stability Limit of detection Limit of quantification
3	19th June – 6th July 2016 Svalbard Archip. (N)	CNR Arctic Base	• System Firmware • System Electronics • LED, PD lifetime and efficiency • Chemistries for each nutrient Reagent stability Limit of detection Limit of quantification • Analysis rate and time • Fluidics Reading on Chip Material compatibility Fluidic design • Inlet sampling system • Battery life

Table 3. Deployment and testing history of the CS Eutrophication Sensor

Three different sensors (light, temperature and pressure) were also integrated into marine housing (in yellow in figure 12, next page) to assess how the reagent will react under the varying environmental conditions. From this deployment the pumping, tubing and reagent lifetime of each reagent was determined. Further development of the system allowed for testing of the firmware, the electronics, reagent stability and the optical detection system during field deployment 2. Incorporated into a deployable bench top the optical detection device for the system was tested on-board regarding the control of light intensity, via a LED, through a standard voltage resistance circuit as well as through pulse width modulation (PWM). This set up had produced linear results for all nutrients in the laboratory in DCU however on-board testing relieved a substantial increase in sensor drift and a reduction in signal stability. This was partially believed to be due to the power source available. The issues highlighted by field-testing were addressed (see deliverable 5.2) and all

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components from both field deployment 1 and field deployment 2 were incorporated into marine housing for field deployment 3. A CS eutrophication autonomous bench top system and autonomous deployable was tested during field-deployment 3 here the autonomous system was used to validate the deployable system. During the deployment issues were observed at the inlet system and in the deployable systems firmware which were addressed and changes made to the system for field deployment 4.

3.3.2 *Exhaustive analysis of the data collected during field testing activities including intercomparisons with commercial sensors;*

Test Site 1

Location: Hawai'i Institute of Marine Biology (Hawaii, USA)
Platform: Floating Pontoon
Components Tested:

- Linear dosing pumps.
- Epi-pump peristaltic pump.
- Diaphragm pump.
- Commercial temperature, pressure and humidity sensor.

Figures 12 and 13 illustrate the set up for field-testing for the three pumps and the sensors for the data logger. The marinised sampling unit is a pelicase with 6 black bulkhead connectors attaching the pump tubes to the box for both the inlet and outlet. Also encased in the marine housing were 3 temperature sensors and one pressure sensor. The first temperature sensor measures environmental temperature and pressure within the box. The second temperature sensor was placed between two of the fluid bags to evaluate how the fluid behaves under different environmental conditions. The third temperature sensor is an inferred temperature sensor, which was also used to determine the effect of temperature on a specific reagent.

Fig. 12. Pump and reagent test system

Fig. 13. System drawing in marine waters

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An LDR (light dependent resistor) was also used to record when the box was opened and closed. All sensor data as well as opening/closing events were automatically recorded with a date/time stamp to an SD card. Further details regarding this test and deployment can be found in deliverables D5.2 for nutrients and D7.2 for the heavy metals precompetitive prototypes.

Test Site 2

Location: Mediterranean Sea
Platform: On-board Wet lab bench top system
Components Tested: Benchtop prototype nutrient sensing system
Date of the cruise: 25 November 2015 – 14 December 2015

- 13 persons for the crew
- 13 researchers
- IMO: 9262077
- Type: RESEARCH/SURVEY VESSEL
- Gross Tonnage: 609
- Summer DWT: 700 t
- Length × Width overall: 46.6 m × 9 m
- Draught: 4.6 m
- Build: 2003
- Flag: ITALY

Apart from the testing activities of the COMMON SENSE sensors, the cruises was also used for the six-month maintenance of five deep moored platforms, three of which are available for COMMON SENSE testing activities: one in the Corsica Channel and two in the Sicily Channel. The vessel and the cruise map detailing the sample sites can be seen in figures 14 and 15 below.

Fig. 14. RV Minerva Uno

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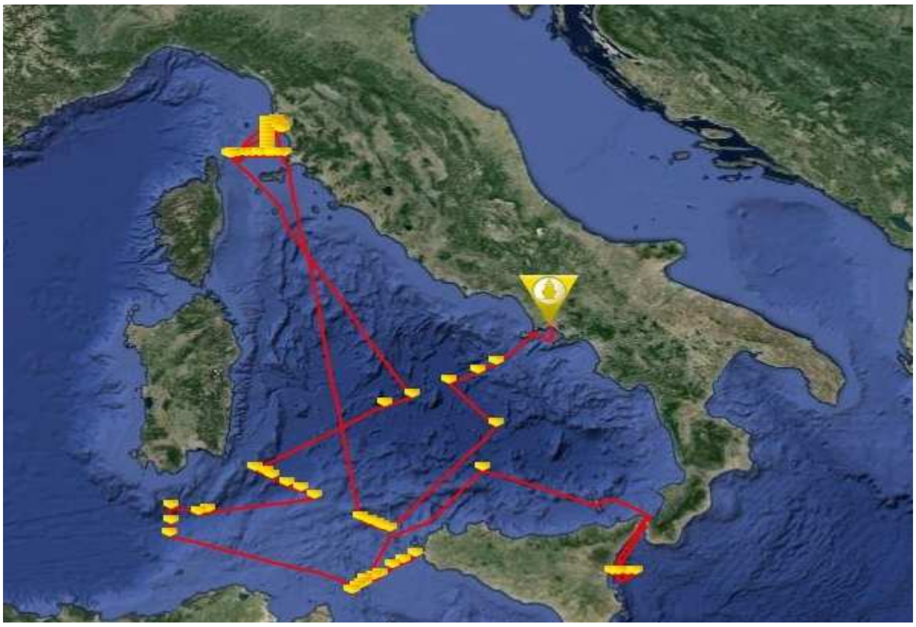

Fig. 15. Map of samplings stations

Water samples have been collected at depths planned during the downcast of the rosette along a vertical profile through the use of 24 and 12 litre Niskin bottles on a General Oceanics rosette (figure 16) equipped with a multiparametric probe and other oceanographic instruments. Initially 20 litres were taken at each depth at the first 5 sampling stations. This was then reduced to 5 litres. These samples were used to test sensors on-board the vessel and by CNR for comparable nutrients analysis. Aliquots of each sample were frozen for subsequent analysis by DCU to further validate the prototype. The system is based on a combination of microfluidic analytical systems, colorimetric reagent chemistry, and low-cost LED-based optical detection. The CS autonomous sensor was tested on-board the RV Minerva Uno for the detection of nutrients by utilising colorimetric chemistries nitrite, nitrate and phosphate. Images from the on-board testing activates can be seen in figure 17 below.

Fig. 16. General Oceanics Rosette Sampler

The COMMON SENSE project has received funding from the European Union's Seventh Framework Program (Ocean 2013-2) under the grant agreement no 614155.

Fig. 17. Water samples being collected from the rosette and filtered for analysis on the CS system

Over 80 samples were analysed on board for nitrite and nitrate, limitations with the system encountered on-board did not allow for the determination of phosphate. The system can be seen as used during on board testing on the Minerva Uno in Fig. **18** below. Approximately, a further 400 samples were taken back to DCU for further analysis.

Fig. 18. Bench top system in use on board the R/V Minerva Uno during field testing

The optical system was tested on-board regarding the control of light intensity, via an LED, through a standard voltage resistance circuit as well as through pulse width modulation (PWM) in a constant current circuit. In order to achieve lower light intensities in the short term the circuit was changed from the constant current set up to a standard voltage resistance set up with no PWM. Here LED intensity was controlled via a 1 kOhm potentiometer. This set up had produced linear results in the laboratory, however this circuit type does suffer from a drift in the sensor signal. On-board testing relieved a substantial increase in sensor drift and a reduction in signal stability. This was partially believed to be due to the power source available. The detection of phosphate is dependent on PWM functionality in order to drive the Ultra Violet (UV) LEDs correctly and so once the circuit was altered to the voltage resistance set up, the system was no longer capable of phosphate detection.

The testing period proved invaluable to the further development of the sensor system as opportunities for improvements to further advance the prototype were highlighted throughout and are addressed on return to DCU and were implemented in the next deployment.

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Figures 19 and 21 show results for nitrite and nitrate produced by the CS bench top system while on board the R/V Minerva Uno. Nitrate and nitrite samples were detectable while phosphate samples shown were not detected were not due to the issues highlighted above. Phosphate results shown in figures 20 and 22 were measured on return to DCU when modifications were made to the CS system.

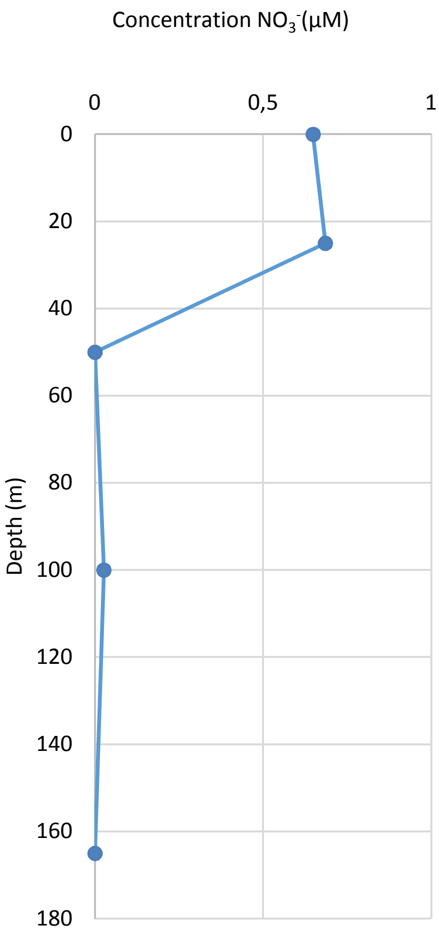

Fig. 19. Nitrate concentrations along a depth profile from 0 to 166m at station 432

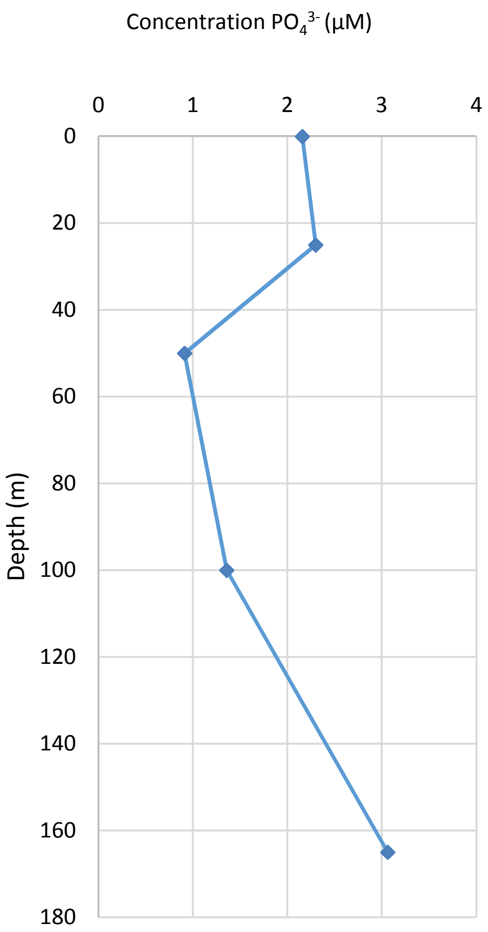

Fig. 20. Phosphate concentrations along a depth profile from 0 to 166m at station 432

Test Site 3

Location: Ny-Alesund, Svalbard, Norway
Platform: Boat, CNR Italy Arctic Base Research laboratory
Components Tested: Benchtop prototype nutrient sensing system and deployable nutrient system
Date: June 2016

Samples were taken at varying stations and depths determined by CNR Italy in Kongsfjorden (see figure 23). Sample were taken using Niskin sampler and analysed at the Italian research base laboratory for phosphate nitrite and nitrate. Nutrient concentrations were determined using the COMMON SENSE deployable systems (figures 24 and 25).

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The CS deployable nutrient sensor (visible in the black box in figure 25) was validated in the laboratory at the research base and deployed on the boat during the last day of the testing period. In 5 stations, named from 001 to 005, samples were taken to measure nutrient concentrations; all samples were analysed using the CS nutrient bench top system. An aliquot of each sample was frozen and returned to DCU and analysed for phosphate nitrate and nitrite on a UV-Visible Spectrometer and the CS benchtop and deployable system. During laboratory and field-testing on site issues were observed with the inlet system. The purpose of the inlet system is to draw sample from the environment and store it within a reservoir, in this case a sample bag. Sample within the reservoir is to be drawn into the nutrient detection system to be analysed. The inlet system must purge dry between each run in order to collect a new representative sample in the reservoir. However unlike testes done in the laboratory during the deployment the reservoir was seen to not fill and retain sample, as the inlet flow rate was equal to the outlet flow rate of the sample bag. As a result there was not enough sample for the pump to draw into the system, this also introduced air into the fluidic and detection system air causing air locks in the system leading to inaccuracy in results obtained during the deployment. The issue was resolved on return to DCU by reducing the reservoir outlet flow rate using different combinations of connections, tubing reductions and increased length of tubing.

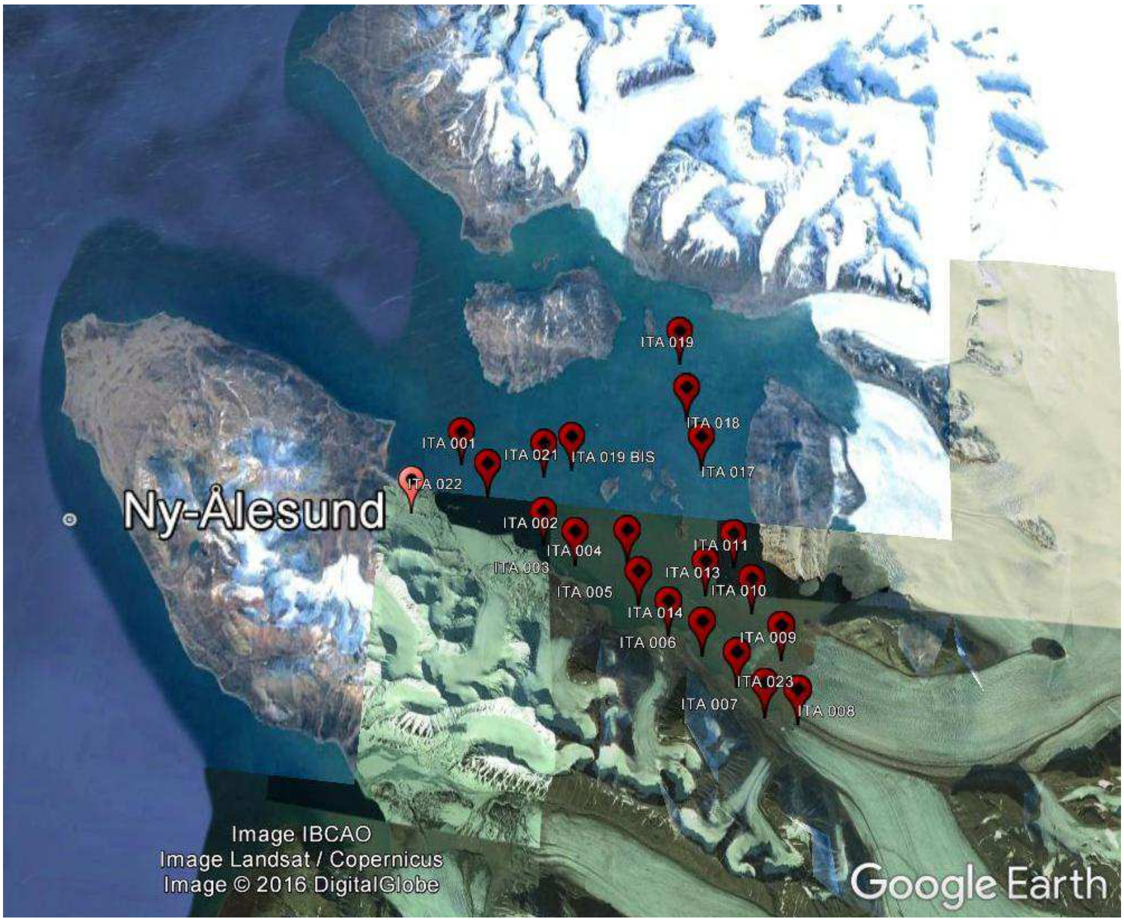

Fig. 23. Map of the sampling stations in the Kongsfjorden, Svalbard, Norway

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Fig. 24. Deployable bench top system acquiring data from samples taken from Kongsfjorden Fjord

Fig. 25. Deployable CS nutrient sensor acquiring samples from Kronebreen Kongvegen glacier

3.3.3 *Short description of stakeholders involved in testing activities with feedbacks;*

The tests at the Hawai'i Institute of Marine Biology (USA) has been realized in the framework of the Alliance for Coastal Technologies (http://www.act-us.info/nutrients-challenge) created with the aim to join the effort to develop affordable, accurate and reliable nutrient sensors.

3.3.4 *Possible optimization actions for prototypes design and performances after testing activities;*

Reduction of analysis time: Currently the system takes sample every 80 minutes further optimisation of the fluidics and the chemistries used in the current prototype could reduce the sampling time and increase the number of samples analysed.

3.3.5 *Limits and optimal installations or possible improvements and definition of further possible optimization actions.*

Inlet filter system for biofouling: The current system requires an inlet filtration system to stop suspended solids entering the system. The filter membranes show evidence of fouling after a period of 19 days. After this time period the fouling starts to affect the performance of deployable system and requires the user to go out and change the filter membrane. The development and integration of an UV-LED based anti fouling module would increase the deployable time and long-term performance of the system.

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3.4 Microplastics - WP6 (LEITAT)

The microplastics sensor is operationally automated to minimize human activities, but is not submersible, through a **camera** and related developed **image processing algorithms** that detect and quantify microplastics particles. It integrates a **water sampling system** with additional discrete sensors like turbidity, fluorescence, CTD, pH, DO_2 and is usable up to 100 m depth. Meaning of these sensors, not being part of the COMMONSENSE project development, is to collect references data to confirm acquisitions from the innovative sensors developed by partners in case of common deployment like temperature, pH, pressure, and to provide complementary information like salinity, chlorophyll-a concentrations and turbidity for the other sensors (nutrients, trace metals, etc).
The sampling system moreover integrates 5 x 0.8 litres Niskin bottles and a submersible peristaltic pump. While the submersible pump flushes the sample through the linked devices, the Niskin bottles are used to collect samples for comparative laboratory analysis and data validation. Devices interoperate thanks to the built-in NMEA data communication protocol developed in collaboration with the project partners. Thorough the data protocol it is possible to acquire data in real time, activate the peristaltic pump or collect a sample using the installed bottles.

3.4.1 *Protocols history of sensor/s;*

Initial version:

The initial idea was to create a multi-channel system based on optical absorption in the near infrared for Fourier Transform Infrared Spectroscopy (FTIR) to detect the microplastic (Refer to D6.1). Due the absorption and attenuation of visible and near-infrared light by sea water, FTIR method was considered not feasible for real time on-situ measurements.

Lab version

Solution (exposed in D6.2) is based on Fluorescence Detector, a type of Fluorescence spectroscopy, which is used for qualification and detection the different type of microplastics by fluorescence profile. The working principle involves using a certain frequency of light (ultraviolet light), that excites the electrons in microplastics of certain compounds, and causes them to emit light, typically visible light. The Camera takes the fluorescence image, and using image processing algorithms the system is able to detect and quantify the particles.
Lab version of the optical camera to test that the field of view was adequate and that the working distances of the illumination and camera were the same, providing a uniform illumination across the whole field of view.

First functional version

Housed prototype simulating standalone real environment operation. Used to validate software algorithms. Change in the tilt of the LEDs and the working distance to improve illumination uniformity on the cuvette. Integration on microplastics sensor

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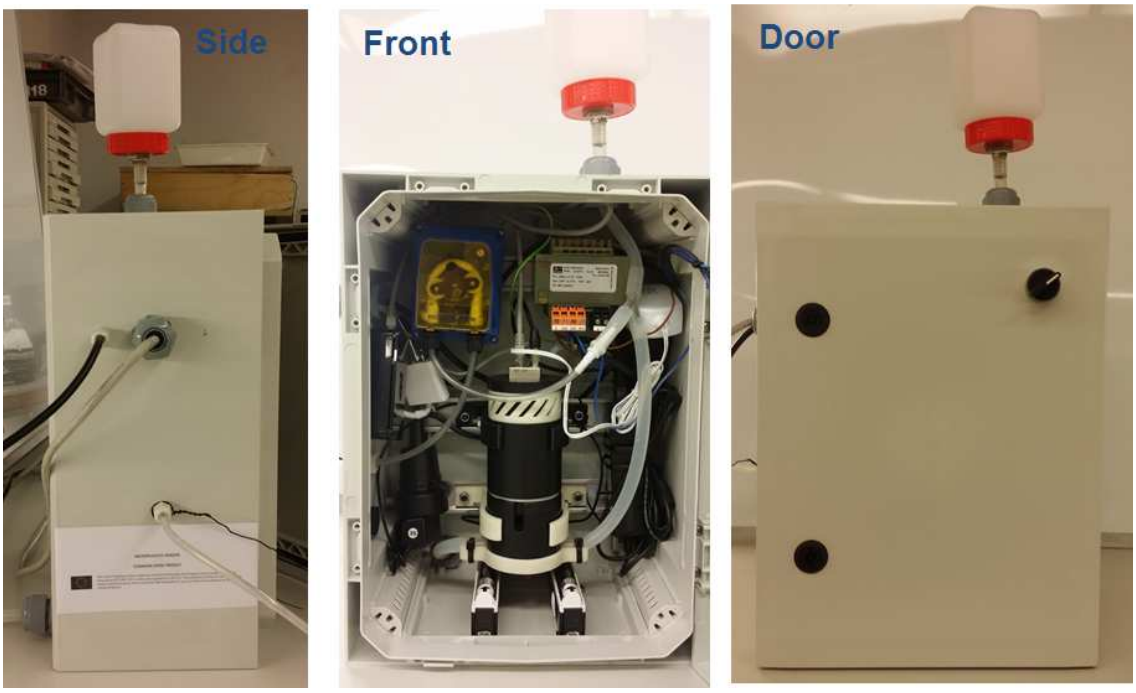

Fig. 26. First functional prototype of microplastic sensor

Second functional version

In order to test in real environment, and considering power requirements, the MP Analyzer is further improved with two types of power supplies (24VDC or 220VAC). Communication with SSU and MP analyser, and related NMEA sequences, have been implemented.

Furthermore, the MP analyzer has changed the light source from UV lamp to UV Led ring to improve ruggedness and electrical isolation and adds a LED strength adjuster. The light transmission mode has been changed to the reflection mode.

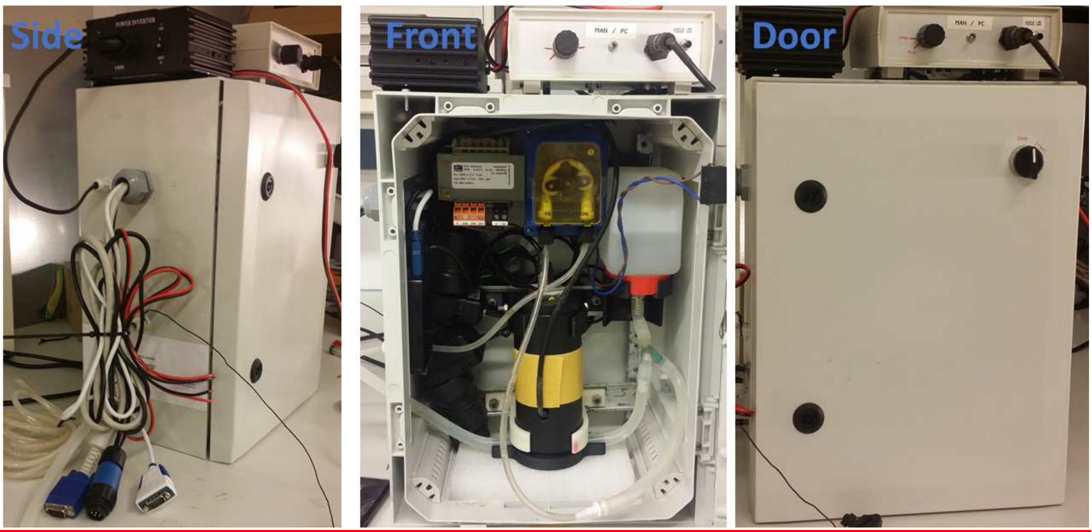

Fig. 27.Second functional prototype of microplastic sensor

The COMMON SENSE project has received funding from the European Union's Seventh Framework Program (Ocean 2013-2) under the grant agreement no 614155.

Vendée globle version

Power converters modified in order to adapt the deployment on IMOCA for VG event (only has 24VDC power supply), which can reduce half of the power consumption.

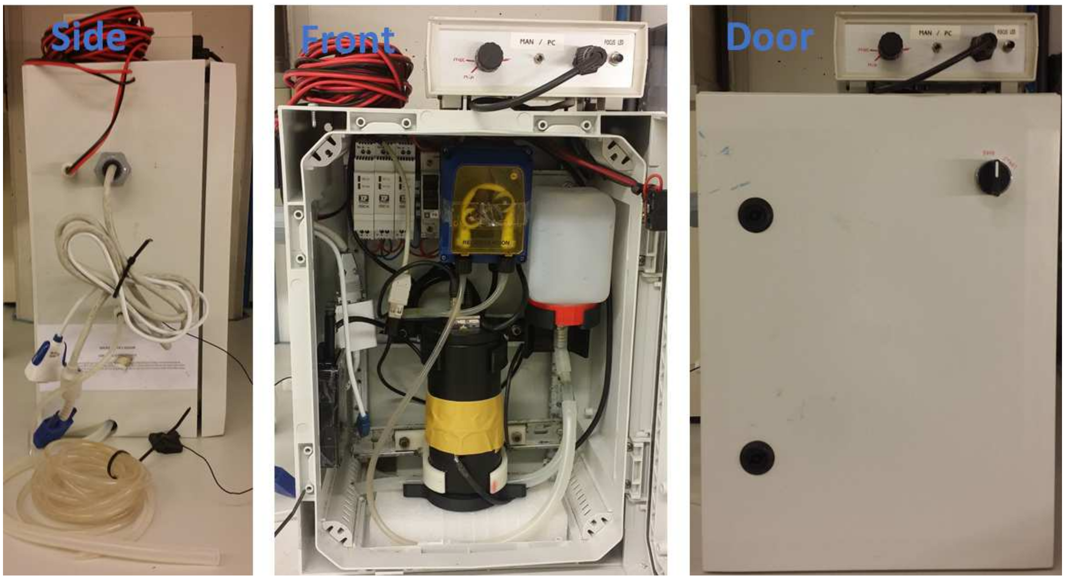

Fig. 28. Vendeé Globe version of microplastic sensor

Below, test activities will be further described. Before any of them, a quick checking was done. Please refer to Annex 6 for detailed information.

3.4.2 *Exhaustive analysis of the data collected during field testing activities including intercomparisons with commercial sensors;*

<u>I testing</u> - IDRONAUT/CNR 25th November – 14th December 2015:

MISS System test – Minerva UNO Research Vessel

The MISS system autonomous profiling capabilities have been tested during a cruise undertaken on board of the MINERVA UNO ship (SO.PRO.MAR. S.p.A. chartered to C.N.R.) from 25 November 2015 up to 14 December 2015.

Figure 29 below shows an example of data acquired by the MISS system during the cruise. The MISS system has been tested at two different stations immediately after the cast with CTD and at 5 different depths between 100 m and surface for two reasons:

- verify the quality of the acquired data through comparison with standard sensors,
- verify the functioning of the water sampler at different depths.

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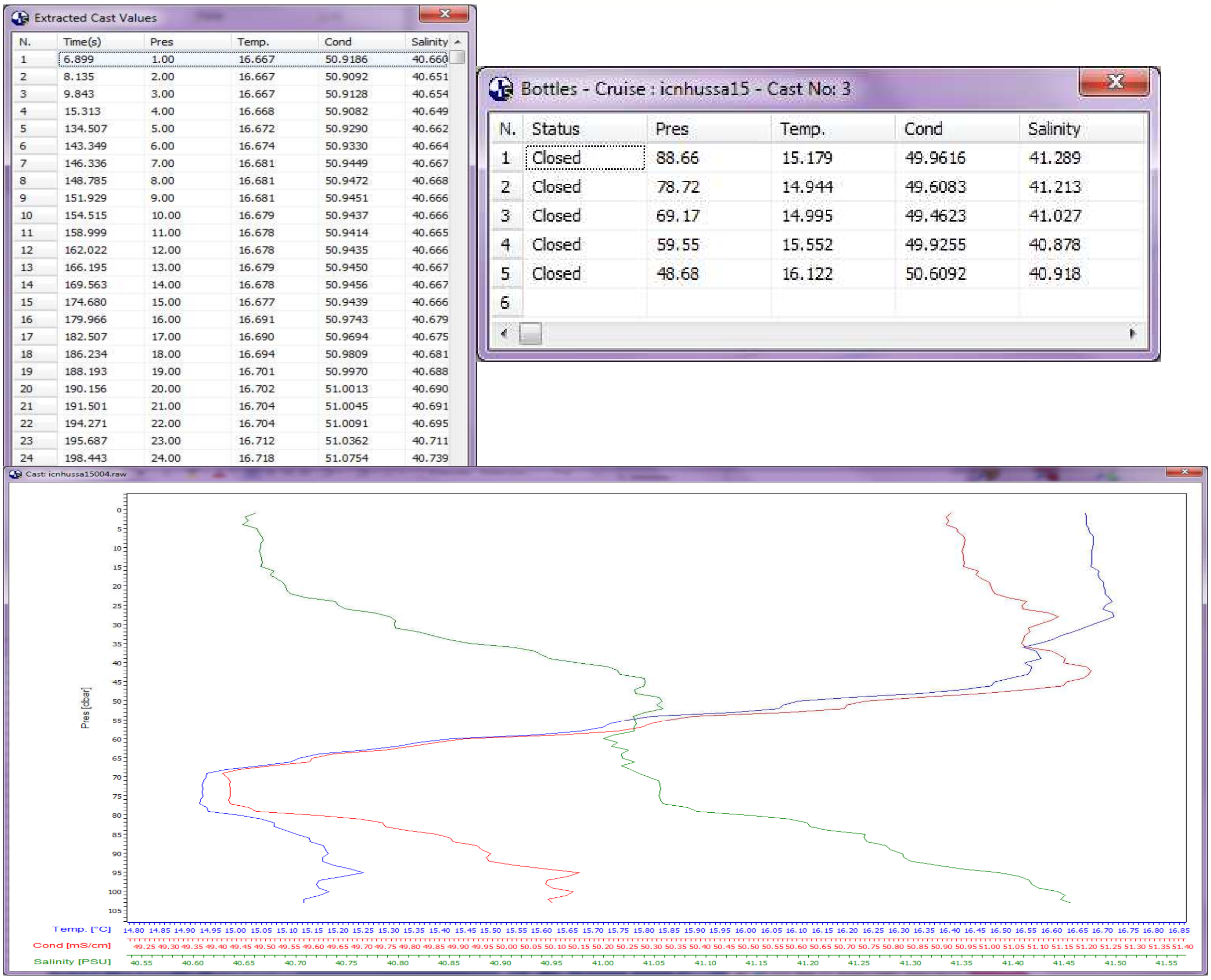

Fig. 29. Results on first testing activity

<u>II testing</u> - LEITAT/FNOB May 14th 2016

Main activity was to test operation of mechanical components and power consumption of developed system, in order to install and deploy it on IMOCA vessel. After fix small issues, a very short term deployment was done on Barcelona coastal area.

This cruise, on board of IMOCA racing yacht (FNOB) has two main aims:

- To test sensor integration for further deployments, specially Vendeé Globe
- To test sensor functionality.

In order to check these items, a one-day test in a coastal area was done in May, in the Mediterranean Sea.

Mediterranean Sea Deployment	
Date	14 May 2016
Place	Kingfisher boat
Cruise Test Time	45 minutes
Cruise Route	Reial Club Maritim de Barcelona -- Near Coast and back

Table 4. Mediterranean test description

The COMMON SENSE project has received funding from the European Union’s Seventh Framework Program (Ocean 2013-2) under the grant agreement no 614155.

Fig. 30. Second testing activity

The Microplastic Analyzer autonomously detected and coarsely quantified the microplastics in the water during a cruise on the board of Kingfisher boat, and performed 45 minutes of uninterrupted test near the coast off Barcelona. The measurement was implemented by a short-range real-time method, where all images were stored, in order to determine and validate the operation of the microplastic sensor by the retained sample. Figure 31 shows the data acquired by MP Analyzer during the cruise, where we can see when we have a detection event. The initial image has been captured, and after that, comparison between each acquired image and this reference image (with any particle) has been done. Figures 32 and 33 show examples of the validation by the saved image and figure 34 shows the collected particles during the cruise.

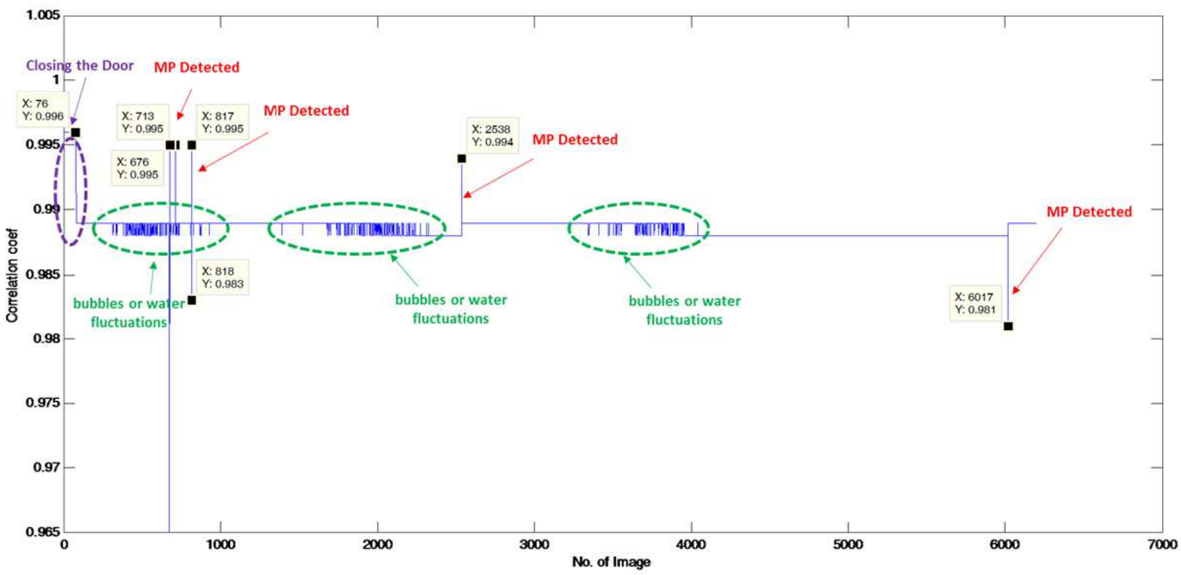

Fig. 31. Correlation coefficients between each image with reference image (first image)

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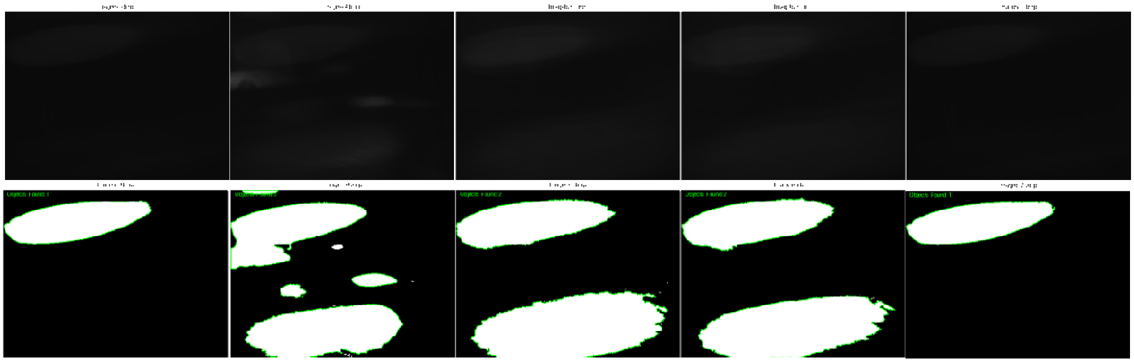

Fig. 32. Detected microplastics particles (Real image—up /Processed image—below)

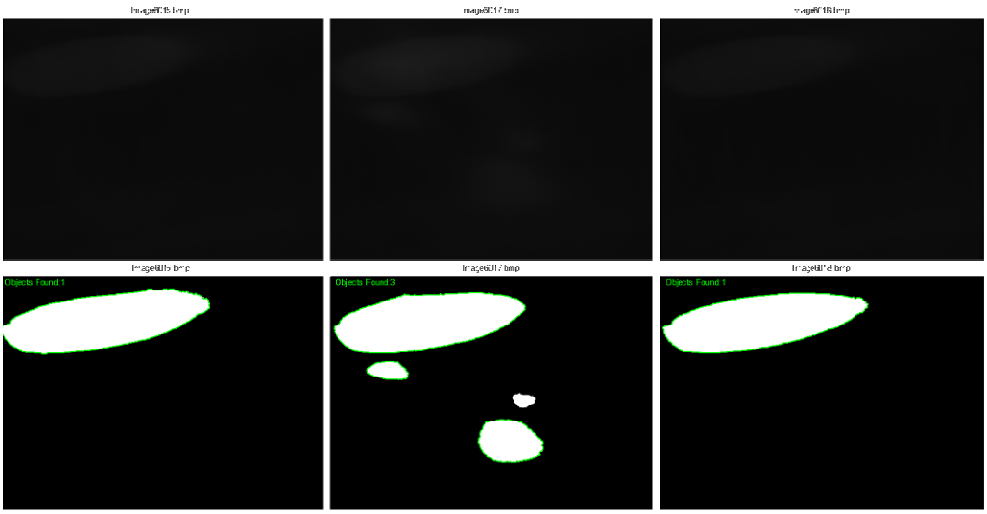

Fig. 33. Detected microplastics (Real image—up /Processed image—below)

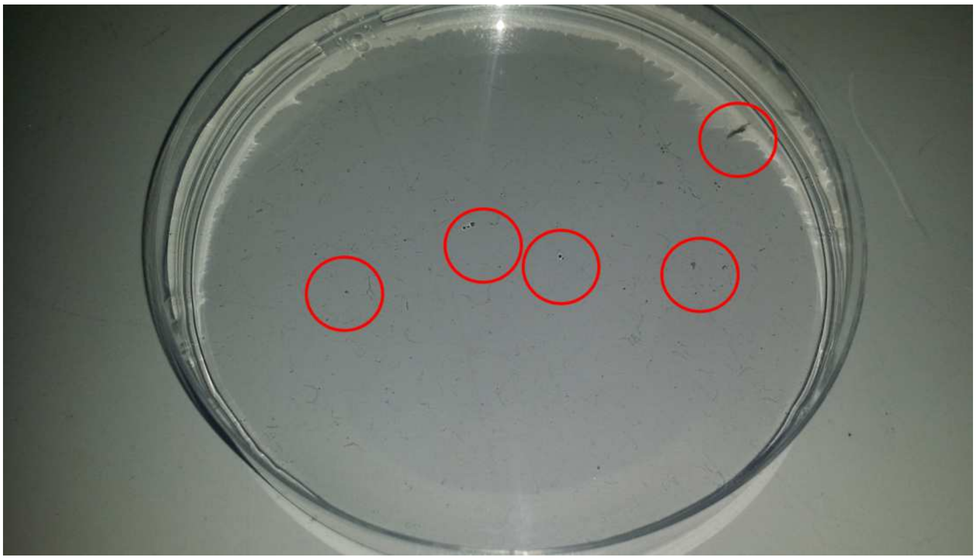

Fig. 34. Collected particles

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The deployment results revealed that the collected values of the correlation coefficient of each image can be used to define the detection and coarse quantification of the MP. The MP Analyzer can save the image automatically by the further validation and fine quantification. However due to the uneven focus of the UV LED, so all the processed images have an oval in the upper-left corner, it did not affect the calculation of correlation coefficient value but it resulted in the false alarm of the bubbles and water fluctuation. Due to the deployment is close to the coast, there are many small micro fibres in the collection. Table 5 summarizes the Mediterranean Sea deployment results.

Experiment Time by Image ID	**Results**
Image ID 673 –677	MP detected
Image ID 712 – 714	MP detected
Image ID 816 –820	No microplastics detection
Image ID 2537 –2540	MP detected
Image ID 6015 –6018	MP detected

Table 5. Mediterranean Sea results

Data reporting

Due extension asked, and accepted on WP4, in order to be aligned with development time of most of the sensors, data interoperation has not yet been tested. In framework of the M30 meeting, in Oristano, one week deployment has been scheduled, where communication between devices will be physically tested and validated.

Communication dataframe and physical specification where defined in previous reports (D6.1 & D6.3, D4.2 and D3.3), On microplastics sensor, this has been implemented and tested versus serial interfaces, but not still under real conditions

Discussion

The after-integration validation tests of the optical camera and optomechanical system have been carried out. One LED was found to be faulty and has been changed, while the effect of the stray light has been temporarily corrected via software so the field testing can proceed while we work on the modifications of the illumination system to suppress this unwanted reflection. All other tests were satisfactory.

We can successfully detected MP during the deployment, it is proven that the algorithms and validation method is correct. However, due the system is very sensitive to the light, the results has impact on the open/close door, and bubbles and water fluctuation, the system require precisely debugging on the focus of light before testing. Since the sampling frequency is very fast (0.5s /image) and the droplet on the quartz tube, there exists quantification error.

On following common deployment (September 2016 in Oristano) data exchange has been tested and validated, furthermore new microplastics sensor detection, quantification, and even the classification has been tested again.

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<u>III testing</u> – LEITAT/IOPAN 13th – 20th June 2016:

The Microplastic Analyzer autonomously detected and coarsely quantified the microplastics in the water during a cruise on board the Polish IOPAN Oceania vessel, and performed 5 days tests during the cruise Gdańsk (Poland) - Trømso (Norway). The idea was to test the microplastics sensor, on board of a research vessel, where human operation and observation is feasible. As shown in the figure below, water introduced by the pump to the vessel, was derived in 4 channels: microplastics sensor, temperature sensor, instrument from eutrophication (out of CS project) and final one to control water pressure. It means that not all acquired water was driven to the microplastics sensor. Anyway, a mesh filter was placed on water output; in order keep all introduced particles, even to the other sensors.

The measurement was implemented by medium or large acquisition, where images were stored only if the event was generated (this means that there was some kind of particle). The validation has been done by the retained sample and saved images:

- Short real time acquisition, where all images were stored, in order to determine and validate real operation of microplastics sensors
- Medium or large acquisition, where images were stored only if event was generated (It means that was presence or some kind of particle)

Baltic Sea Deployment	
Date	13-20 June 2016
Place	OCEANIA Boat
Cruise Test Time	5 days deployment (24 hours / day)
Cruise Route	Gdansk (Poland) – Tromso (Norway)

Table 6. Baltic Sea deployment description

Fig. 35. Set-up of microplastics sensor inside the OCEANIA research vessel

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Fig. 36. Samples filtering for storage and comparison

Table 7 shows the summary of the five days MP detection during the IOPAN cruise. Fig. **42** shows the collected particle during the cruise.

Experiment	Detected Time	Results
Day 2	07:30:21	MP detected
	07:30:30	MP detected
	07:58:53	MP detected
	10:29:58	MP detected
Day 3	10:52:48	MP detected
	14:14:50	MP detected
Day 4	20:44:42	MP detected

Table 7. Baltic Sea results

Detailed Results of Baltic Sea Deployment

Below results from different days are shown, for each day there is an output chart and a table summarising results. The figures below indicate the correlation coefficients between each image with reference image (first image); the title of each figure indicates the testing period.

When the MP is detected, the right side images (the dark ones) shows the detected image with quantitative information. These images are saved only when the parameter indicates that the MP is detected, they are mainly used to validate the parameters.

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Day 1: Tue 14 June 2016

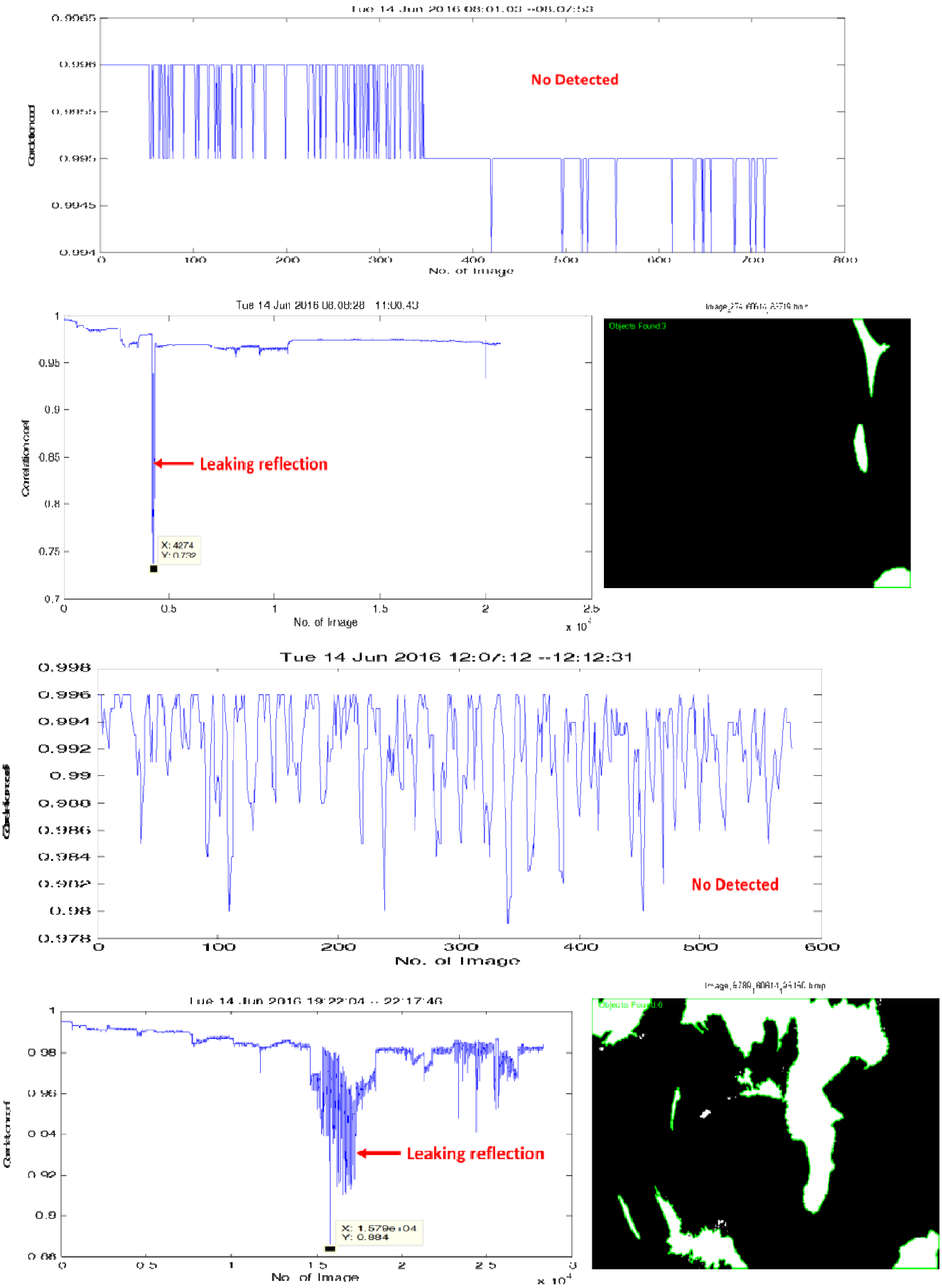

Fig. 37. First day Baltic deployment results

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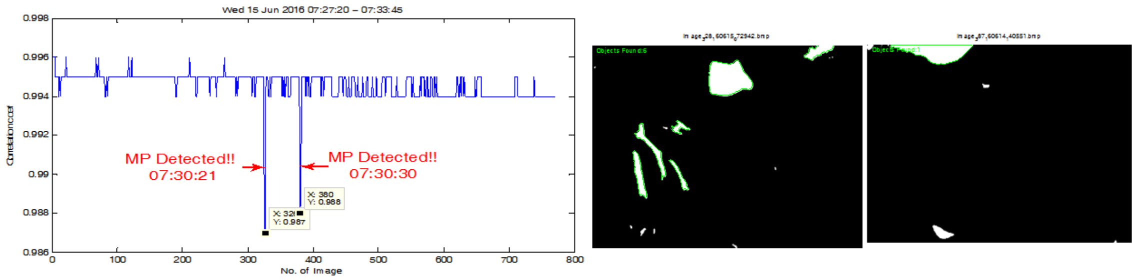
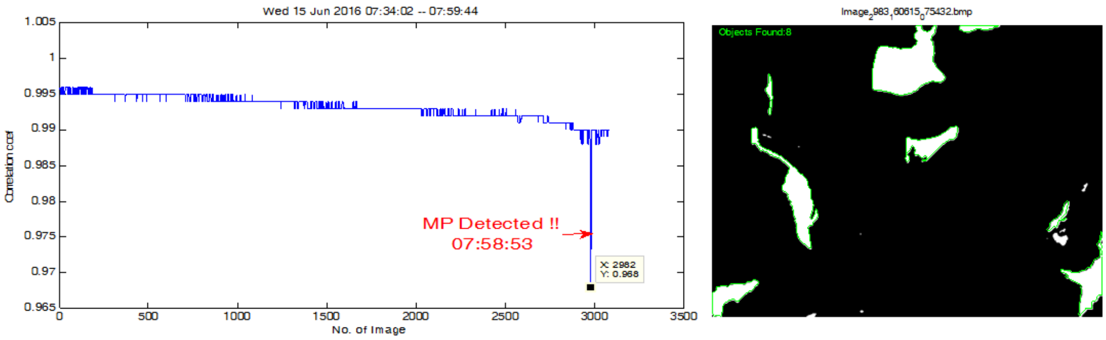
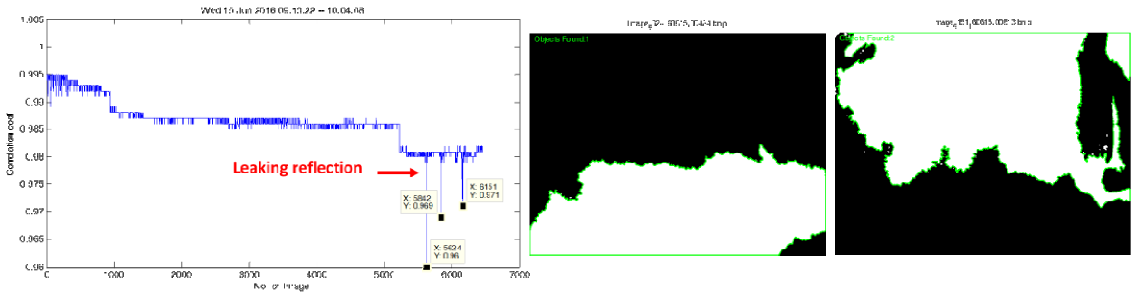
Day 1 Experiment Time	Results
08:01:03 – 08:07:53	No microplastics detection
08:08:28 –11:00:43	No microplastics detection but detect leaking reflection
12:07:12 – 12: 12:31	No microplastics detection
19:22:04 – 22:17:46	No microplastics detection but detect leaking reflection

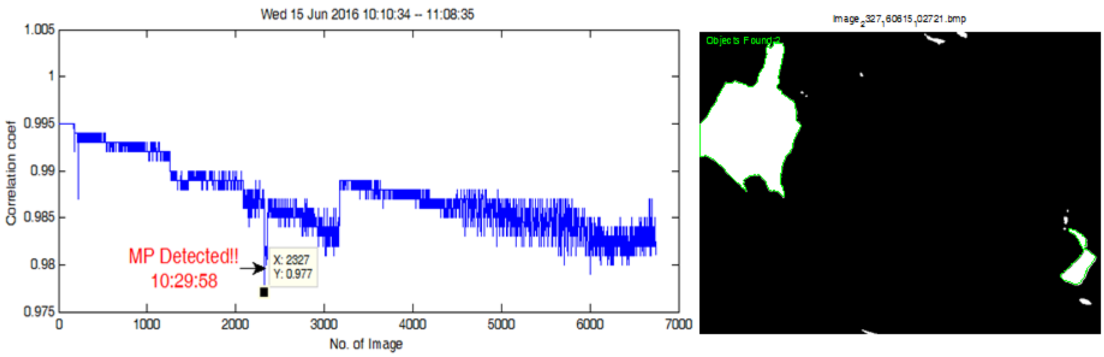
Table 8. First day Baltic deployment results

Day 2: Wed 15 June 2016









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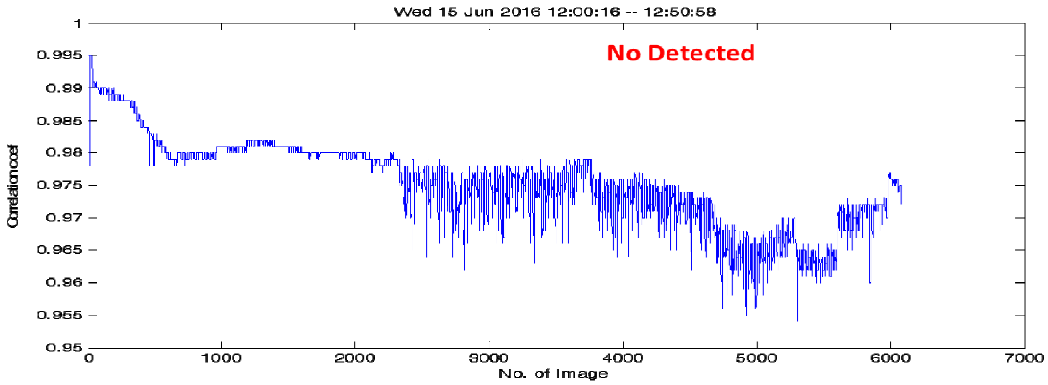

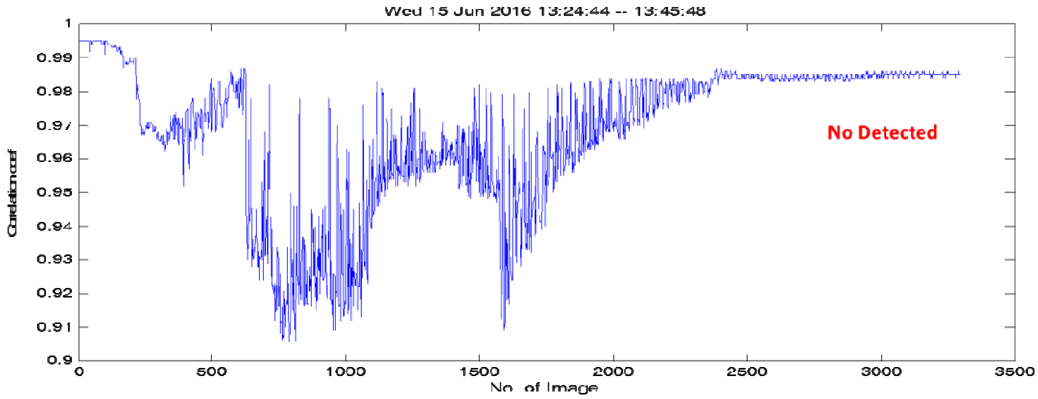

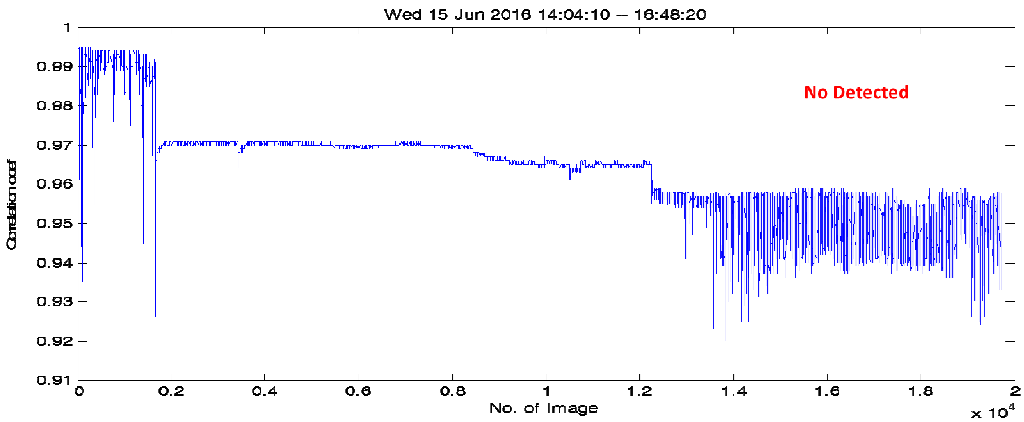

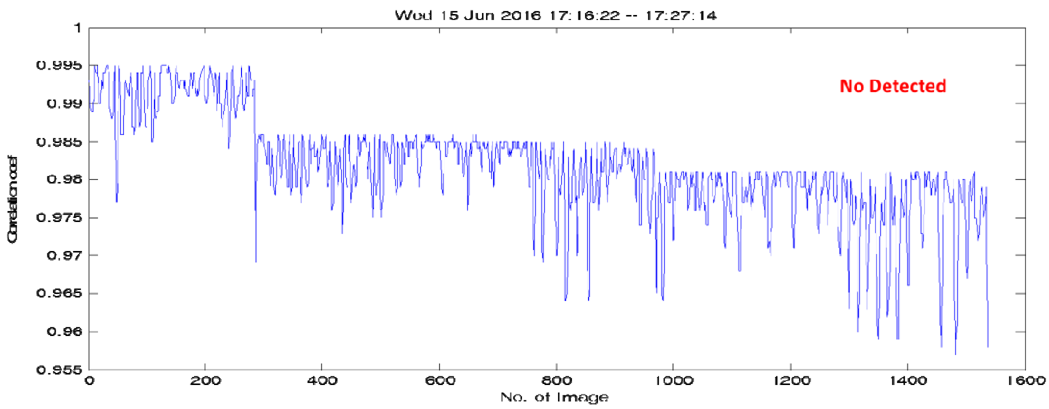

The COMMON SENSE project has received funding from the European Union's Seventh Framework Program (Ocean 2013-2) under the grant agreement no 614155.

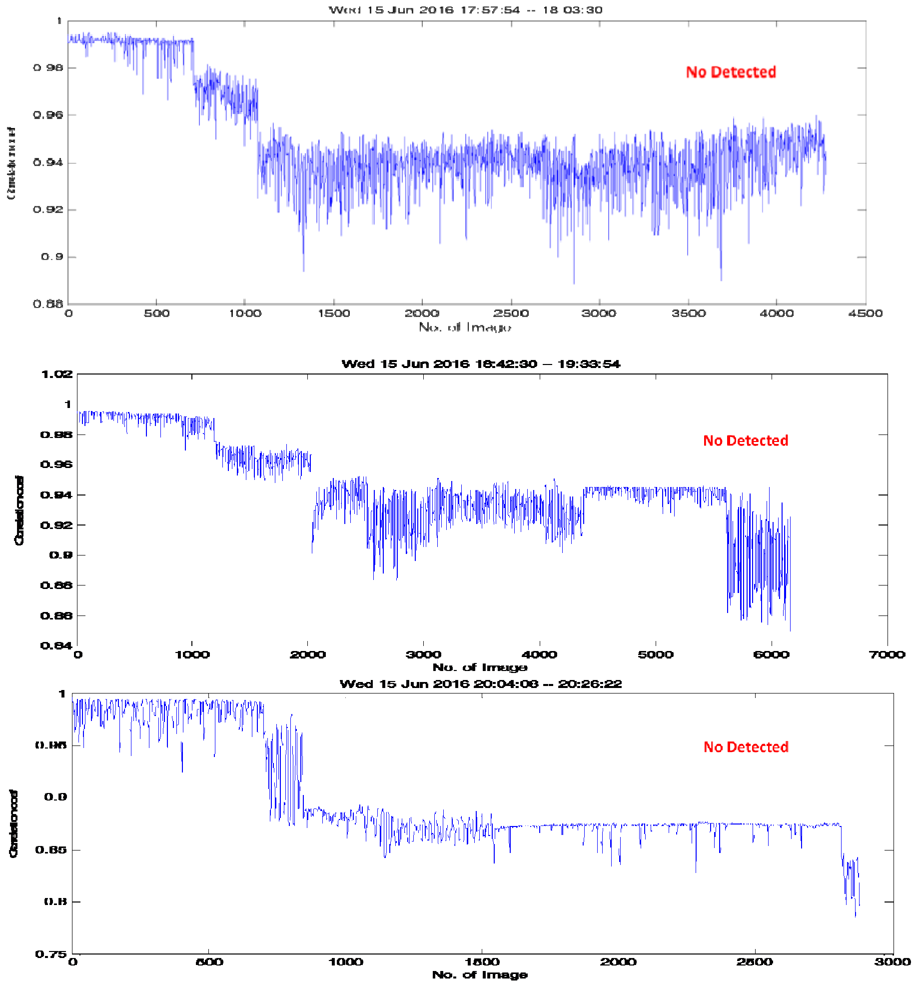

Fig. 38. Second day Baltic deployment results

Day 2 Experiment Time	Results
07:27:20 – 07:33:45	MP detected at 07:30:21; MP detected at 07:30:30
07:34:02 –07:59:44	MP detected at 07:58:53
09:10:22 – 10:04:08	No microplastics detection but detect leaking reflection
10:10:34—11:08:35	MP detected at 10:29:58
12:00:16 – 12:50:58	No microplastics detection
13:24:44 – 13:45:48	No microplastics detection
14:04:10 – 16:48:20	No microplastics detection
17:16:22 – 17:27:14	No microplastics detection
17:57:54 – 18:03:30	No microplastics detection
18:42:30 – 19:33:54	No microplastics detection
20:04:08 – 20:26:22	No microplastics detection

Table 9. Second day Baltic results

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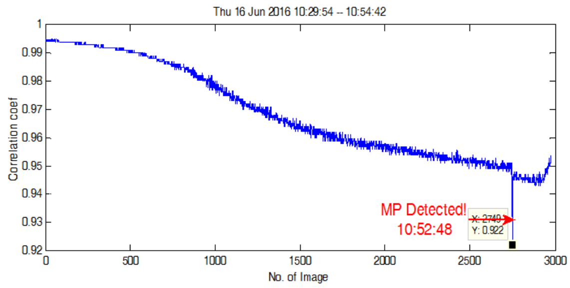
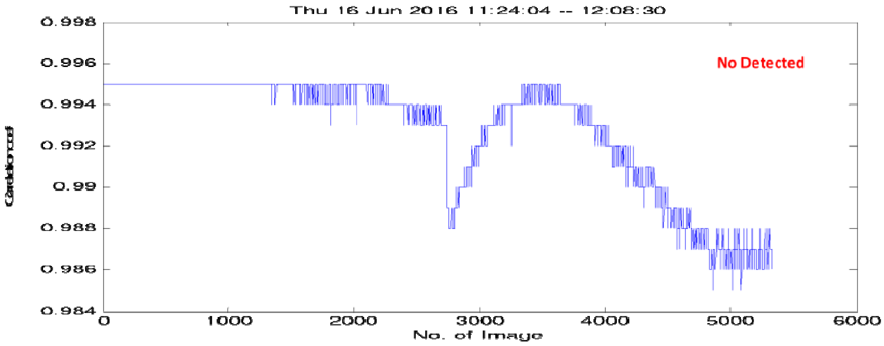
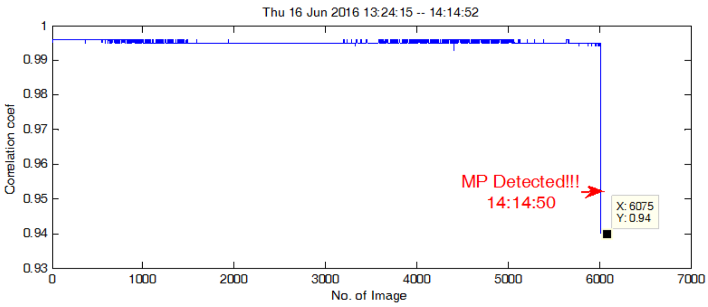
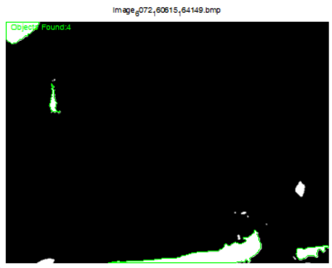
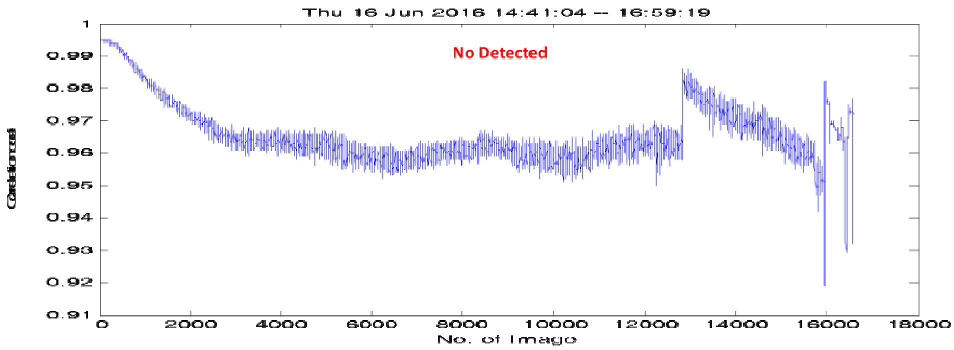
Day 3: Thursday 16 Jun 2016











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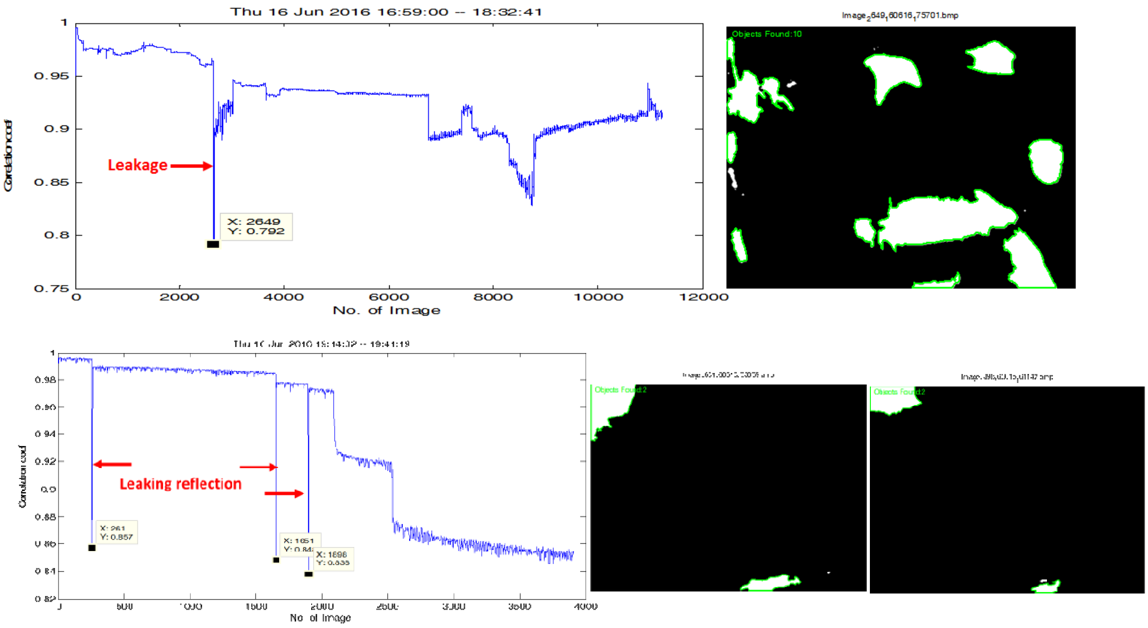
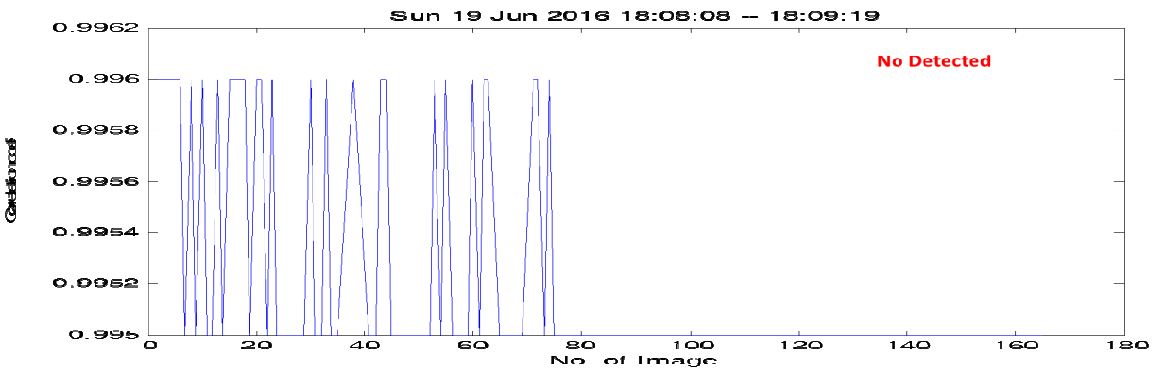


Fig. 39. Third day Baltic deployment results

Day 3 Experiment Time	Results
10:29:54—10:54:42	MP detected at 10:52:48
11:24:04 – 12:08:30	No microplastics detection
13:24:15—14:14:52	MP detected at 14:14:50
14:41:04—16:59:19	No microplastics detection
16:59:00—18:32:41	No microplastics detection but detect leaking reflection
19:14:02—19:41:19	No microplastics detection but detect leaking reflection

Table 10. Third day Baltic results

Day 4: Sunday 19 Jun 2016



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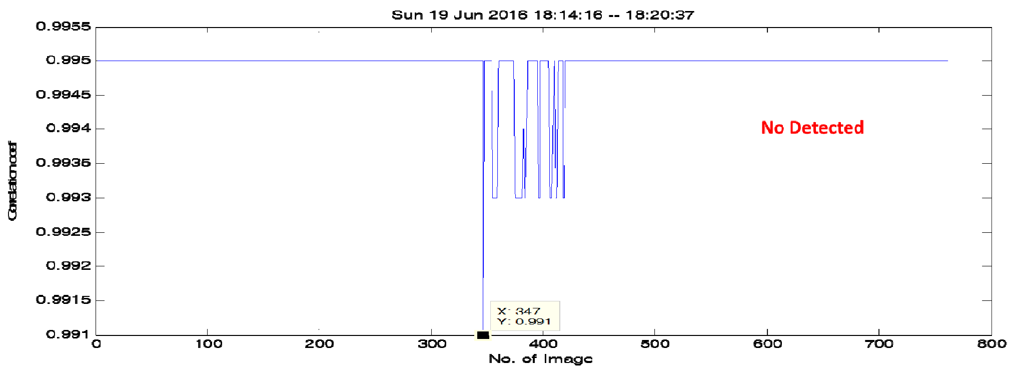

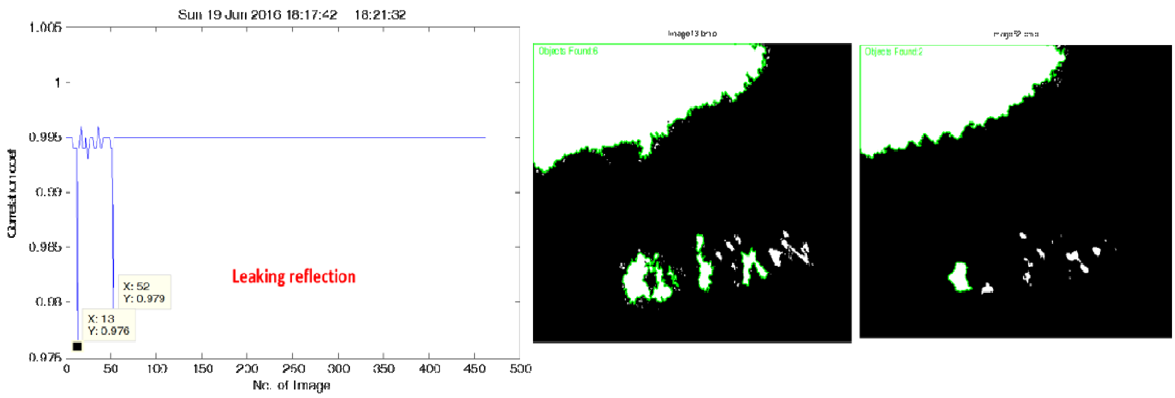

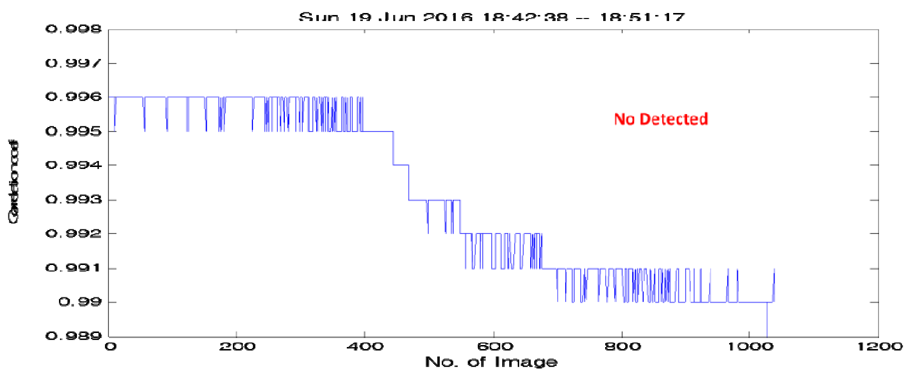

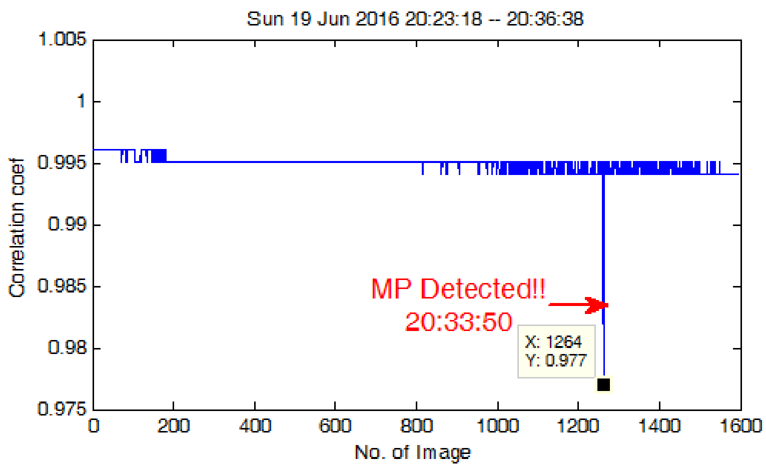

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Fig. 40. Fourth day Baltic deployment results

Day 4 Experiment Time	Results
18:08:08—18:09:19	No microplastics detection
18:14:16—18:20:37	No microplastics detection
18:17:42—18:21:32	No microplastics detection– Leaking reflection
18:42:38—18:51:17	No microplastics detection
20:23:18—20:36:38	MP detected at 20:33:50

Table 11. Fourth day Baltic results

Day 5: Monday 20 Jun 2016

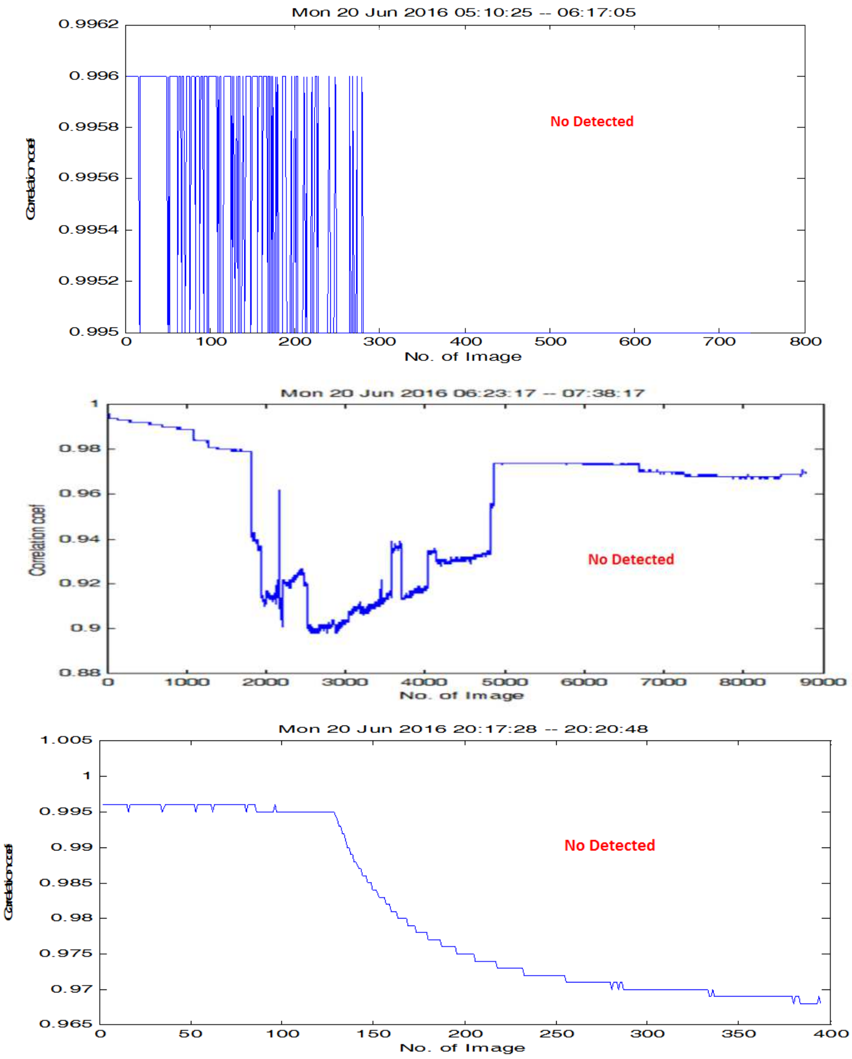

Fig. 41. Fifth day Baltic deployment results

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Day 5 Experiment Time	Results
05:10:25—06:17:05	No microplastics detection
06:23:17—07:38:17	No microplastics detection
20:17:28—20:20:48	No microplastics detection

Table 12. Fifth day Baltic results

Figure 42 below shows the collected particles by the Microplastic Analyser during the whole deployment.

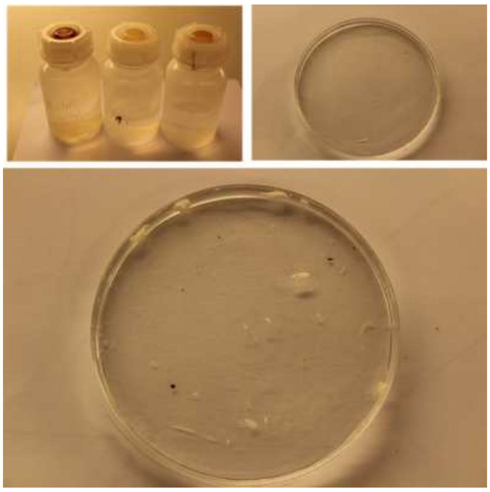

Fig. 42. Acquired samples for different days: sample bottles (upper left), second day (upper right), third-fourth day (below)

Data reporting

Due extension asked, and accepted on WP4, in order to be aligned with development time of most of the sensors, data interoperation has not yet been tested. In the framework of the M30 meeting, in Oristano, one week deployment has been scheduled, where communication between devices will be physically tested and validated.

Communication dataframe and physical specification were defined in previous reports (D6.1 & D6.3, D4.2 and D3.3). For the microplastics sensor, this has been implemented and tested versus serial interfaces, but not yet under real conditions

Discussion

The after-integration validation tests of the optical camera and optomechanical system have been carried out. One LED was found to be faulty and has been changed, while the effect of the stray light has been temporarily corrected via software so the field testing can proceed while we work on the modifications of the illumination system to suppress this unwanted reflection. All other tests were satisfactory.

We have successfully detected MP during the two deployments, it is proven that the algorithms and validation method is correct. However due to the water leak on the quartz tube, and uneven exposure to UV LED, resulting in several false alarms which we did not expect during the Lab validation. Since the sampling frequency is very fast (0.5 s/image) and the droplet on the quartz tube,

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there exists quantification error. Since the system is very sensitive to the light, the results has impact on the open/close door, and bubbles and water fluctuation, the system require precisely debugging on the focus of light before testing.
On following common deployment (September 2016 in Oristano) data exchange has been tested and validated, furthermore new microplastics sensor detection, quantification, and even the classification has been tested again.

Parameter	MISS System	SBE CTD	DESCRIPTION
Temperature (°C)	21.792	21.897	Installation
pH	8.068	7.972	Installation
Conductivity (mS/cm)	47.554	47.980	Installation
Salinity (PSU)	65.188	65.861	Installation
Temperature (°C)	21.800	21.898	Bottle #1
pH	8.068	7.972	Bottle #1
Conductivity (mS/cm)	47.546	47.980	Bottle #1
Salinity (PSU)	65.201	65.861	Bottle #1
Temperature (°C)	22.174	22.025	Bottle #2
pH	8.068	7.969	Bottle #2
Conductivity (mS/cm)	48.061	48.263	Bottle #2
Salinity (PSU)	66.322	66.372	Bottle #2
Temperature (°C)	24.094	24.097	Bottle #3
pH	8.095	8.032	Bottle #3
Conductivity (mS/cm)	48.507	48.547	Bottle #3
Salinity (PSU)	69.486	69.550	Bottle #3
Temperature (°C)	24.271	24.283	Bottle #4
pH	8.110	8.040	Bottle #4
Conductivity (mS/cm)	48.479	48.226	Bottle #4
Salinity (PSU)	69.695	69.402	Bottle #4
Temperature (°C)	24.275	24.143	Bottle #5
pH	8.112	8.028	Bottle #5
Conductivity (mS/cm)	48.508	48.528	Bottle #5
Salinity (PSU)	69.736	69.589	Bottle #5

Table 13. The most significative parameters acquired during the MISS installation and at the time a bottle has been closed

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IV testing – IDRONAUT/LEITAT/CNR 26th – 28th September 2016: Analyzer and MISS System were deployed at CNR laboratories and in the Mistral lagoon close to the CNR institute in ORISTANO (I). Integration with SSU and MISS was tested and validated. There was no microplastics detected because most of the measurements were performed in a filtered tank.

Then MISS system sampling and data acquisition capabilities have been tested in the Mistral lagoon. The MISS system was directly connected and controlled from the micro-plastic analyser. During the field installation data was collected on a 5 minutes time basis for all the installation period. The 5x0.8l Niskin bottles have been collected time to time for the successive laboratory comparative analysis.

The MISS deployment allows to acquire on a time basis (5 minute) reference physical, chemical and optical data about master sea-water parameters, from a traditional CTD installed on the MISS system and to collect five (5) 0.8 liter samples for comparative laboratory analysis. The below table reports a summary of collected data and the master sea-water parameter associated to each sample. MISS data can be compared with a second CTD (SBE) installed by the CNR aside of the MISS System.

The complete set of acquired data has been loaded on the COMMONSENSE web OGC data portal. Herewith in table 13, in the previous page, the most significative parameters acquired during the MISS installation and at the time a bottle has been closed are reported.

V testing – LEITAT/FNOB 6th November 2016:

Microplastics sensor is installed on IMOCA racing yacht, which is participating at the Vendeé Globe event during the preparation of this report. Once participation will be over, data will be recovered and further analyzed and if is possible, due timeline, we will add this information to final report.

3.4.3 *Short description of stakeholders involved in testing activities with feedbacks;*

Due the state of the art, stakeholders were not involved within tests, even if after presentation on different events (Oceanology International or BOOS conference) opportunities to do that appeared. We concentrate all the efforts in testing it within the Consortium.

Fishery at Mistras lagoon (Oristano) have been really interested in collaboration for the field testing of all sensors realized in the COMMON SENSE, permitting the use of their facilities, but they didn't participate directly as they just asked for the project and the developed sensors.

3.4.4 *Possible optimization actions for prototypes design and performances after testing activities;*

Improvement of the electrical isolation of the LED ring: this has already been implemented, as it was found earlier during testing that the LEDs cathodes and anodes were not sufficiently separated when soldered to the ring and some LEDs started switching on and off due to electrical discharges.

During the field trials it was confirmed that the light emission remains constant within the needs of the detection software. The detection algorithm works as well in the lab as when the power is supplied by the boat itself. However, as is explained in section 3.4.5, it was found that the electrical isolation of the LEDs cathode and anode was not sufficient. The necessary modifications have already been implemented.

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Strengthening of the LED ring: since the prototype will be tested for three months during the submission of this report in a racing yacht and will therefore be subjected to rough seas, it was decided to make the LED ring more ruggedized for it to survive to the trip. The necessary modifications have already been implemented.

The deployment results reveal that the MP analyser has successfully detected MP during two deployments. The sampling frequency is 2Hz, with resolution 2592x1944 pixels. It can work automatically or controlled by the commands from SSU. However due to reflected light on the Quartz tube, the bubbles and water fluctuation can result in several false alarms and quantification errors.

Several approaches were used to try to get rid of the unwanted reflection on the cuvette surface, like installing bandpass filters in front of the camera optics to get rid of the direct reflection. However, these approaches were unsuccessful, and the correction by software remains at this time the only feasible alternative. A new solution, definitive is proposed in section 3.4.5, but it is expensive and could not be undertaken before installing the prototype on the Imoca vessel.

Due to the perspective of lighting (position and direction), there are some bias in the MP quantification and detection by bulb. The water flow and image processing system is applicable by the experiment, but the processing time needs to be improved.

The MISS sampling system may be optimized by increasing the size of the Niskin bottles from the present 0.8 litres up to 2.0 litres.

The lab experiments proofs that the UV and violet light (320 and 400 nm) can be used to detect 60% of less denser microplastic, certain pigments can be detected, depending on the material of the pigments. The MP detection will not be affected by sands, algae and water. Actions to improve this percentage should be take into account.

3.4.5 *Limits and optimal installations or possible improvements and definition of further possible optimization actions.*

Due to characteristics of microplastics acquisition, nowadays is not efficient to measure in a consistent way microplastics at sea. Current methods use nets filtering more than 1 million litres, to get a significant sample. In this sense, the main issue which needs to be solved is the **acquisition and pre-processing system**. Although the sampling system is working perfectly, it needs to take more water flux in order to acquire significant sample volumes. This high volume implies modifications on the data processing software, as well.

In this sense, the change of the water pump for another sampling system, or the use of a higher volume pump plus filtering stage should be considered.

In order to improve the optical part of the microplastic analyser is mandatory to eliminate detrimental reflections on the cuvette surface totally. In order to do this, it would be necessary to change the cuvette geometry. Indeed, its cylindrical cross-section makes it unavoidable that a part of the light will be reflected by the cuvette surface towards the camera, as exemplified in the following figure.

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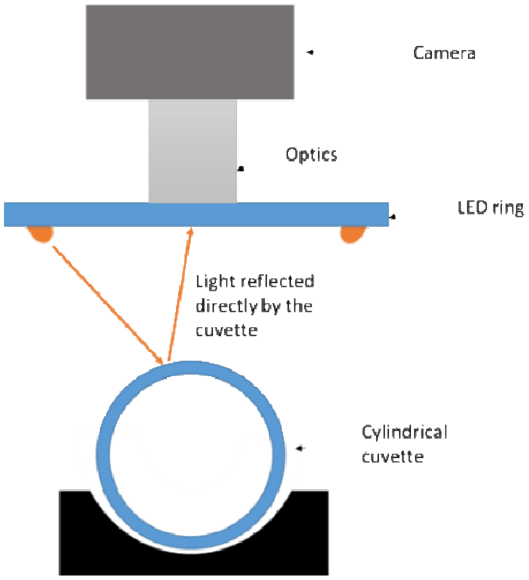

Fig. 43. Schematic drawing of the current prototype configuration

This detrimental reflection is made even worse by the fact that, due to the tilt of the LEDs this reflection falls well within the field of view of the camera.

A plane-parallel cuvette, on the other hand, would reflect light towards the camera at a much lower angle, as shown in the following figure. It would therefore be much easier to diaphragm the light adequately so to eliminate reflections totally within the field view of the camera.

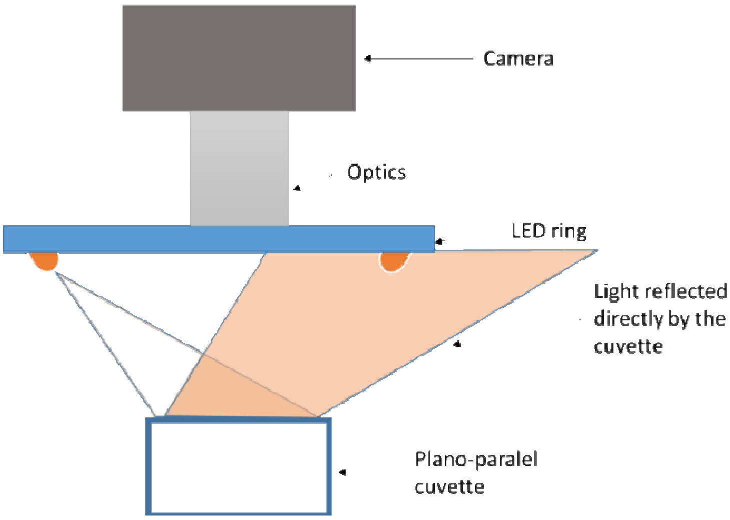

Fig. 44. Schematic representation of the suggested modification (with a plane-parallel cuvette and properly diaphragmed illumination)

In this case, the most important factor would be to find an adequate combination of cuvette and working distance so that the corners are just outside the field of view, because here it is where the worst reflections would occur.

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3.5 Heavy metals - WP7 (DROPSENS)

3.5.1 Protocols history of sensor/s;

Autonomous system for "in situ" analysis of Pb(II), Cd(II). Cu(II) and Hg(II). The system includes a microfluidic chip for complete mixing of the buffer and the sample, a potentiostat to perform electrochemical measurements, two peristaltic pumps to control the fluidic pathway through the system, a wall-jet flow cell which incorporates the screen printed electrode (sensor). The electrode has been manufactured using a new developed carbon bismuth nanoparticle composite ink, for the determination of Cd(II) and Pb(II) and a new developed carbon porous material ink for the determination of Cu(II) and Hg(II). The validation of the sensor has been performed with artificial samples and natural seawater, as explained in D7.3. Briefly, artificial samples where used to optimize the parameters of the square wave technique and to evaluate the response of the electrodes to the different heavy metals. Carbon bismuth electrodes have shown a linear response in the range 5 – 100 ppb for the determination of Cd(II) and Pb(II) and carbon electrodes have shown a linear response in the range 5 – 70 ppb for the determination of Cu(II). Carbon material with gold nanoparticles electrodeposited in its surface have been tested for Hg(II) determination, with a linear response in the range 1 – 10ppb. Reproducibility between electrodes has been demonstrated for all the heavy metals mentioned above.

Validation of the sensor using natural seawater samples has been performed in the "Institut de Ciéncias del Mar" (ICM-CSIC) in the aquaria and experimental chambers facilities (Barcelona). The water is obtained from an underwater intake 300 m off coast at 10 m depth. The facility is computer controlled and monitored 24 hours a day. Testing of Cd(II) and Pb(II) was performed "in situ" in the flume chamber taking samples from a tank containing "old" water (filtered seawater that was left there for more than one month) and another containing "new" water (water recently pumped without any treatment). The results showed that neither of the samples tested had significant values of Cd(II) and Pb(II). The sample were spiked with a mixture of Cd(II) and Pb(II) in concentration 25 ppb each. The electrode could detect both analytes simultaneously. The detection of Cu(II) in "old" seawater samples was positive. The peak was identified adding known concentrations of Cu(II) in the sample. It was concluded that the electrodes worked correctly for the detection of Cd(II), Pb(II) and Cu(II).

The results obtained were in accordance with the periodic control analysis carried out in the ICM-CSIC. Once the validation of the sensor has been performed, the prototype system has been validated in the ICM-CSIC laboratory (Barcelona, Spain) and in the DCU laboratory (Dublin, Ireland). Optimization of parameters was necessary in order to have the system ready for field-testing activities.

3.5.2 Exhaustive analysis of the data collected during field testing activities including intercomparisons with commercial sensors;

Once all of the individual components had been tested and determined, the precompetitive prototype system could be formally designed. The microfluidic chip, fluid bags, pumps, tubes and

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connectors tested throughout work package WP7 were combined into a single hydraulic system, designed around the requirements set out by the project partners. A flow through reservoir style system has been chosen for incorporation into the system design. This sample fills a reservoir from which the less powerful but more accurate peristaltic pumps can draw from and pass through to the chip and electrode. When it is time to collect a new sample the inlet pump runs for a time to completely fill the reservoir again with fresh sample water, the previous unused sample water is displaced and passes through the outlet, exiting the system. This ensures that each time a sample is collected the system is drawing from a fresh source and is not simply retesting the previous sample liquid. It also reduces the dead volume in the sample tubes.

Once the parts were selected and the fluid path defined, the physical system could be designed, incorporating the electronics, the potentiostat, the fluidics into the marinised housing a more detailed overview of the deployable system is found in D7.2 and D7.3.The system uses the same type of environmental enclosure as it was trialled during the field testing outlined in paragraph 3.3 on sensor for eutrophication, however in a smaller size, the bulkhead connectors for the sample inlet and outlet are also the same as those trialled. Initial tests of the deployable system took place on-board the RV Minerva Uno where field-testing proved to be vital in the further development of the deployable system.

Test Site 1

Location: Mediterranean Sea
Platform: On-board Wet lab bench top system
Components Tested: Benchtop prototype nutrient sensing system
Date of the cruise: 25 November 2015 – 14 December 2015

Fig. 45. Heavy Metals System Gen 1 on-board the RV Minerva Uno

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The precompetitive prototype was tested in the field as part of work package 9 on-board the R/V Minerva Uno Ichnussa cruise 2015 as part of WP9. During the 21 day research cruise the following components were tested:

1. Peristaltic pumps
2. Stepper motor drivers
3. Tube materials (Tygon, Viton and PTFE)
4. Tube connectors
5. Flow through cell
6. Reagent storage containers

The following issues were encountered and addressed on return to DCU:

1. Flow through pump head observed while motors were not in use:

Two solenoid valves were added to the fluidic path, their inclusion deemed necessary following the field-testing period aboard the RV Minerva Uno in the Mediterranean Sea. The constant motion induced a slight flow through the pumps while not in use. As a result these active valves were added to ensure there was no flow through the system while the pumps were off.

2. Stepper motor drivers became hot:

The control boards used to drive the stepper motors became hot after prolonged use. While this was not a concern given the integrated thermal shutdown, it did indicate a more power efficient solution might be available. Disabling the control boards when not in use and thereby reducing their energy consumption achieved this.

3. Flow through cell:

Fluid was observed to leak out of the flow through cell from where the electrode is inserted. It was found that clamping the cell sides together with greater force could prevent this leak. A mechanical latch was designed and added to the cell which once closed applied the correct clamping force and prevented further leaks.

4. Reagent storage containers:

Ridged containers and flexible bio processing bags were tested for their ease of use and manual handling. Ridged containers were found to be easiest to handle given their fixed shape, however these would require a pressure equalisation valve in order to allow fluid to be drawn from them. Conversely the flexible bio processing bags require no such valve given they simply deform as they are emptied, however they proved to be difficult to handle as they had no fixed shape. The solution was the use of commercially available flexible cube shaped fluid storage containers (1 l LDPE Cubetainer). These containers are easier to handle than bio processing bags as they are stiffer and retain a more solid shape, however they remain flexible and so no vacuum release valve is required in order to draw fluid from them.

Mixing of the reagent and sample is performed by passing both fluids simultaneously through a microfluidic chip. The serpentine channel inside the chip forces both fluids into the same path and mixes them through subjecting the fluid to variable shear as it passes through variable radius curves.

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This is a similar chip designed for WP5 however it is only used for mixing in this case, the optical detection path is not required as the heavy metals sensor is housed in its own flow through cell after the mixing chip. Initial trials were conducted without the use of a mixing chip by pre-mixing the sample and reagent and passing this fluid directly to the electrode for measurement. The flow rate, pump timings and valve actuation is governed by the microcontroller, automating the fluid handling system.
A schematic of the fluid handling system is shown in figure 46.

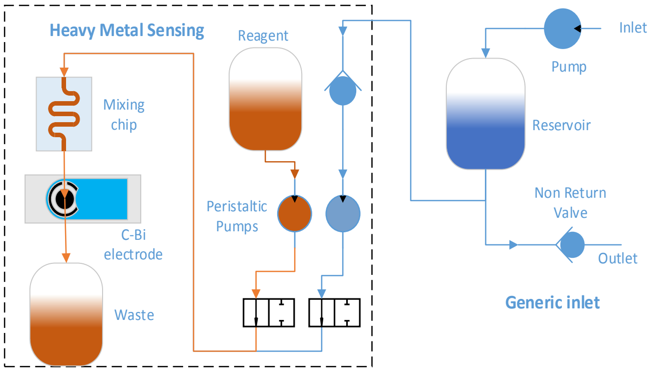

Fig. 46. Fluid schematic of the version 2 heavy metal system

Detection system/ Flow through cell:
This portion of the system is comprised of the screen printed electrode, the DropSens electrode cable connecting the electrode to the potentiostat and a modified DropSens flow through cell which houses the electrode.
The flow through cell was modified to include a mechanical latch used to clamp both sides together under greater force as some leaks were detected during the field trial on-board the R/V Minerva Uno.

Electronic control, data acquisition system, data storage and communication modules:
Electronic control of the fluid handling system is governed by the microcontroller.
This runs the pumps at a given flow rate by turning the motor by a precise angle, one step at a time at a set speed.
By simultaneously turning both motors through the same angle at the same speed the pump heads deliver an equal volume of fluid. Both pumps can by run simultaneously for mixing or individually if required.
Data acquisition is performed by the commercially available DropSens potentiostat contained within the pre competitive prototype, which is captured and saved by the accompanying laptop.

Power supply:
As noted in the technical report for the first reporting period (M18), power is supplied via mains supply on-board the vessel following WP discussions with DropSens and CSIC during the M12 and M18 partner meetings establishing this as the preferred deployment platform.

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Marinized Housing:

Several changes were made to the internal chassis used for testing the version 1 prototype on-board the R/V Minerva Uno. The original chassis was manufactured from 4 mm thick laser cut acrylic sheets, reinforced to 8 mm thick in key areas. While this chassis design did survive the field testing period a redesign was deemed necessary in order to create a stronger more robust system for use in the marine environment. For the version 2 system, structural acrylic components were replaced with 1.5 mm thick 316 stainless steel sheet.

This provided a much stronger frame allowing all components to be securely bolted in place. This particular steel type is corrosion resistant and is the preferred grade for use in the marine environment.

All bolts, screws and nuts were restricted to this steel type to help reduce the chances of corrosion.
Acrylic sheets were still used for low force and non-structural components. The reasoning behind this design choice was the ease of manufacture and assembly.
Such components include the reagent storage rack and the splash guard covering the electronics.
The external housing chosen for the system was a pelicase.
These cases are commercially available and have been used in previous testing periods with great success affording the internal components protection against liquid ingress and physical impacts. This can be seen in figure 47 below.

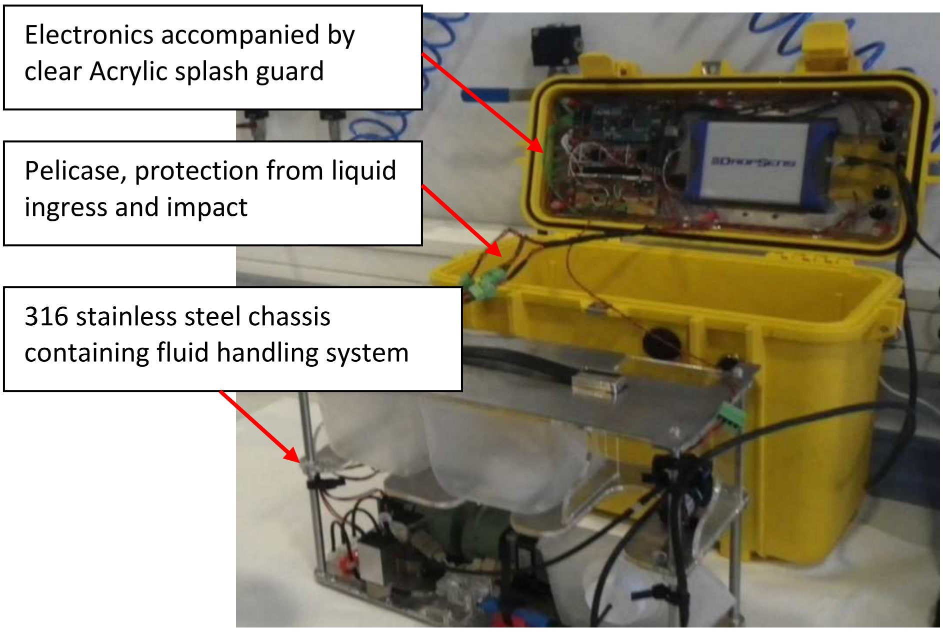

Fig. 47. Heavy Metals System Gen 2: New fluidic design incorporated into a Pelicase waterproof housing and 316 stainless steel internal chassis

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Figure 48 shows the newly designed fluidic system for heavy metals determination after the redesign and fabrication of the new system.
The system was brought Institute of Microelectronics of Barcelona (IMB-CNM-CSIC) for full validation and testing. Reagents used for Validation were provided by TELLAB.

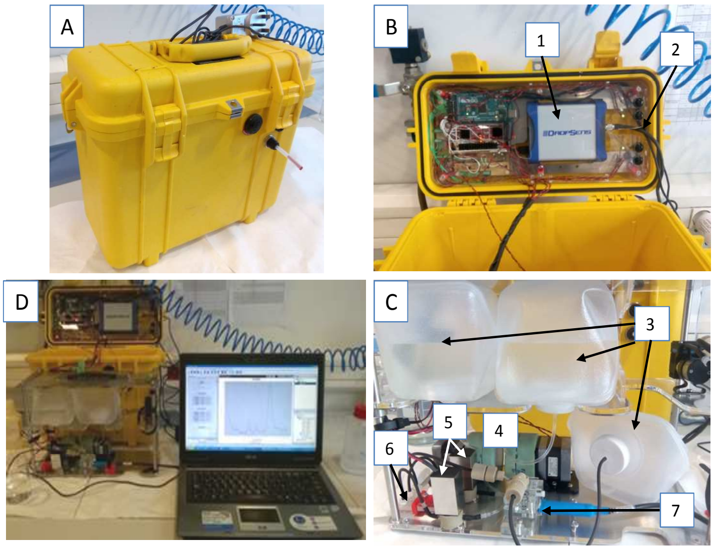

Fig. 48. Heavy Metals System Gen 3 A) Marine housing containing the fluidic system prototype. B) Electronic system of the prototype including 1) the potentiostat and 2) four bottoms to control the different pumps. C) Fluidic system containing 3) three plastic reservoirs for containing the sample, the buffer and the waste, 4) two peristaltic pumps, 5) two valves, 6) fluidic mixer and 7) wall-jet electrochemical fluidic cell where the screen-printed electrode (SPE) is placed. D) Fluidic system connected to a laptop

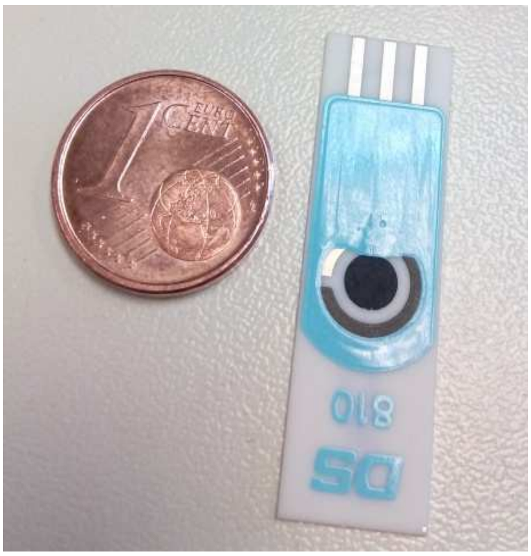

Fig. 49. Electrochemical device.

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Measurements with the redesigned fluidic system:
Voltammetric measurements were performed using the Heavy metals fluidic system Gen2 developed by DCU with commercial screen printed carbon electrodes (SPCEs). The system mixes the buffer (4 ppm Bi in 0.2 M acetate buffer pH 4.5) with the sample (containing Cd and Pb) in a ratio 1:1.

Pump flow rate:
The flow rate of the pumps in the deployable system was evaluated to see the device was constant and the same for both pumps. The measurements were done by weighing the amount of solvent pumped in 30 s, which was collected from the tubing right after the valves that follow each peristaltic pump. It is observed in Table 14c that, the larger the amount of solution in the reservoir (V) is, the larger the amount of solution in 30 s is pumped and, consequently, the faster the flow is. This indicates that the flow is in some extent driven by gravity because the reservoirs are located above the fluidic system.The following issues were encountered and addressed on return to DCU:

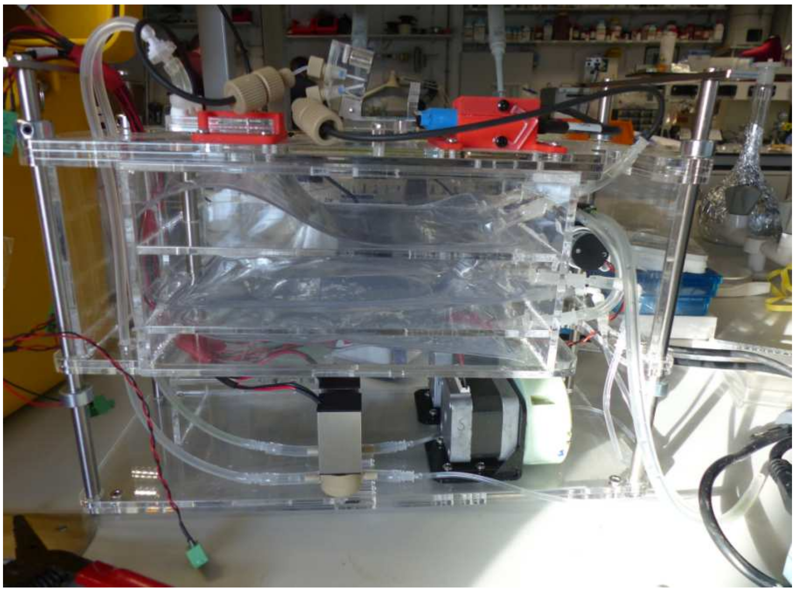

Fig. 50. Heavy Metal System Gen 3

The different problems that were faced during this month working with the prototype are the following:

- The problem with the formation of a bubble in the wall-jet electrochemical chamber is still an issue. Although it is possible to eliminate it as it was it is sometimes not so easy and it takes longer to remove it. Sample bags have been changed holders have been designed to hold the bags at an angle to illuminate air bubble generated in the fluid reservoirs from entering the system.
- The formation of bubbles in the tubing mainly occurred when the solutions of the reservoirs were changed. A purge steep has been incorporated to illuminate air in the sample tubing

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- An accumulation time of 300 s is necessary to detect 1 ppm Cd and Pb under constant solution flowing in the wall-jet cell. Changes to the firmware will allow the flow rate on the system to be adjusted to account for the accumulation time on the system.

Solutions cannot be injected in the system when this container is placed below the position of the pump. Sample/Reagent bags have been placed above the pumps an valve placed on the bags to also for solutions to be changed without the bags being removed

1st measurement:			
a) V1 & V1'	Replicas		
"Buffer" pump:	4.8510 g	4.8190 g	4.7855 g
"Sample" pump:	5.2068 g	5.3340 g	5.1684 g
2nd measurements:			
b) V2 & V2'	Replicas		
"Buffer" pump:	5.8022 g	5.4805 g	5.2479 g
"Sample" pump:	6.0146 g	5.7786 g	5.7141 g
3rd measurements:			
c)	V2	V3	V4
"Buffer" pump:	5.2479 g	5.8626 g	6.1485 g

Where: V2<V3<V4

Table 14. Amounts of solvent pumped in 30s by the pumps of the fluidic system

Test Site 2

Location: Ny-Alesund, Svalbard, Norway
Platform: Boat, CNR Italy Arctic Base Research laboratory
Components Tested: Bench top prototype nutrient sensing system and deployable nutrient system
Date: June 2016
Samples were taken at varying stations and depths determined by CNR Italy in the Kongsfjorden (see figure 23). Sample were taken using Niskin samplers and stored for further analysis on the heavy metals system.

3.5.3 *Short description of stakeholders involved in testing activities with feedbacks;*

Fishermen at Mistras lagoon (Oristano) have shown great interest in collaborating in field testing of all sensors realized in the COMMON SENSE project and allowed the use of their facilities.

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For heavy metals, Dublin City University has participated in the field testing activities (Oristano, September 2016).
TelLab was involved providing buffer samples and stock standard solution, DropSens provided electrodes and support with the use of the software that controls the Potentiostat and CSIC validated the methods that were used for the determination of the heavy metals.

3.5.4 *Possible optimization actions for prototypes design and performances after testing activities;*

As explained in paragraph 3.5.2, during field-testing activities performed during the CNR cruise in the western Mediterranean on board the R/V Minerva Uno (25^{th} Nov – 14^{th} Dec 2015) it was necessary to perform some changes in the design of the heavy metals sensor. The new system was validated in the IMB-CNM-CSIC laboratory, showing the need to continue improving the design of the system and the fluidic cell.

3.5.5 *Limits and optimal installations or possible improvements and definition of further possible optimization actions.*

Screen-printed Electrode (SPCE)
The reproducibility of the SPCE in the fluidic system was evaluated, as this would determine the deployment type for the system. Successive electrodes were used to measure different concentrations of Cd and Pb reproducibility between the different measurements was poor indicating that the electrodes would be used once for each measurement. Further optimisation of the electrodes to allow for multiple sample analysis

Installation of LCD screen to control the flow.
The flow of the sensor was fixed during validation activities and was not possible to change it manually during testing activities. To facilitate the control of the flow rate, a LCD screen will be install in the sensor. Using the LCD screen, the user could change the flow rate during analysis of samples. The integration of a LCD screen in the heavy metals sensor will be achieve before December 2016, during the last months of the COMMONSENSE project.

3.6 Underwater noise - WP8 (CEFAS)

3.6.1 *Protocols history of sensor/s;*

Field Deployment #1 – An existing underwater pressure housing was used to deploy the equipment. This enabled easy access for debugging and development of the electronic system. The proposed hydrophone and logging system were in place and configured to capture raw data, which could then be analysed to assess system performance.

Field Deployment #2 – This trial utilised the same system configuration that was deployed in the first field trial, but this time in the Baltic Sea. This enabled us to assess the system performance in an open water environment.

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Field Deployment #3 – This trial included the bespoke housing, reduced power consumption and noise analysis algorithms to derive summary data of 1/3 octave bands. This summary data could not be transmitted as the design used a GPS source, instead of the SubCtech logger.

Field Deployment #4 – The final field trail included integration with the SubCtech logger.

3.6.2 *Exhaustive analysis of the data collected during field testing activities including intercomparisons with commercial sensors;*

The second field trial conducted and reported by IOPAN, included a comparison of the noise sensor with commercially available sensor.

The measurement point was located nearby of the waterway approach to the Gdynia port at a distance of 5 cables northerly to the navigational buoy GD, with coordinates position λ = 45^{O} 32.516'N, φ =018^{O} 37.1'E (position has been designated by the Maritime Administration in Gdynia). Depths at the harbor fairway and beyond are approximately 20.0 m. With the average number of about 20 ships transiting per day, the Gdynia waterway may be counted as a rather low traffic noise area compared to the Western Baltic seaways or the English Channel (one order times higher). Results of one day records clearly show that mean anthropogenic noise can be locally high. It was found that contributions of other sources to underwater ambient noise, as road noise from the Gdynia city, or pile driving in the harbour are likely but supposed to be insignificant compared to the nearby shipping.

The setup was deployed on the sea bottom for about 24 hours, and later for a further 12 hours. The equipment worked perfectly. Due to 24 bit A/C converter the dynamic of recording covered the broad range of noise from ambient noise characteristic or sea state 1 up to nearby passing ships.

A freely floating sphere was attached to the frame, for security reasons. This resulted in a characteristic ringing (disturbance) to be observed in the time series data.

Only after additional spectral analysis characteristic properties of the disturbances were found, a subtraction procedure was developed which almost totally eliminates interference from the frame.

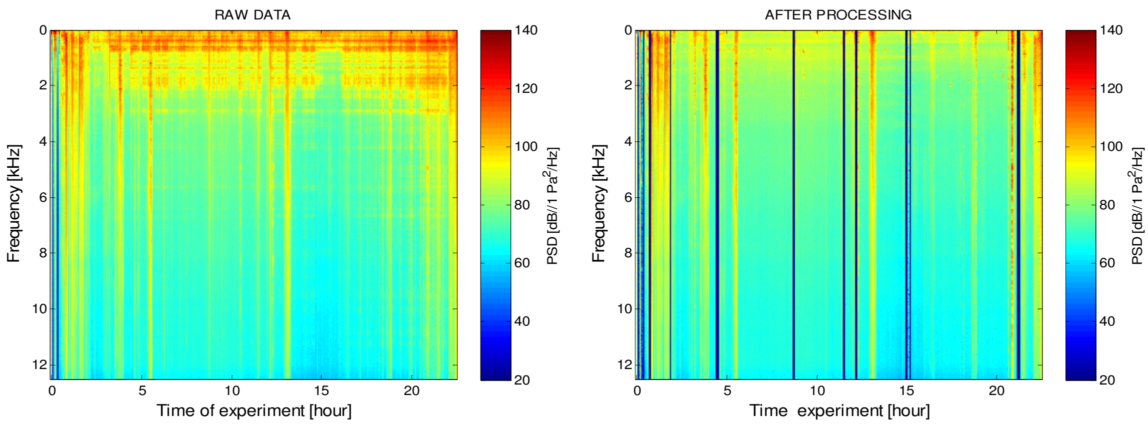

Fig. 51. Power Spectra Density Level of underwater noise before (on the left) and after removing signals with the local disturbances (on the right).

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Due to very low level of electric signals at the output of hydrophones and usually high level of electromagnetic noise from buoy components, it leads to interference of the acoustic signals transformed to electric form, and true electromagnetic signals. The level of electric noise inside of the CEFAS unit was compared with 16-bits and other off-the-shelf passive acoustic buoys. In figure 52 there are presented results of spectral analysis of signals registered by some commercial passive acoustic buoy. We observe multitude of horizontal lines with the level around 40dB on this commercial sensor, which are not present on the COMMONSENSE Noise Recorder.

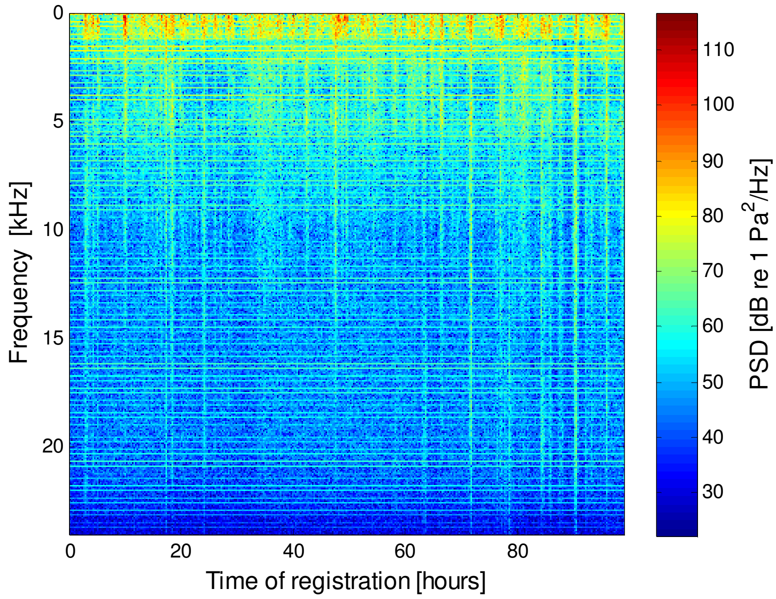

Fig. 52. Spectra of signals registered by commercial buoy, with low sensitive hydrophone.

IOPAN commented "It was found that the CEFAS equipment is of scientific-quality, with expected high dynamics and high signal-to-electric noise ratios".

The final field deployment was conducted in a lagoon at CNR in Oristano during 26-29 September 2016.

The recordings showed variable background noise conditions in the lagoon (figure 53), with possible dusk and dawn choruses apparent at low frequencies, and occasional vessel passages appearing as brief vertical bands of noise. The recordings suffered from tonal noise originating in the 50 Hz mains power supply to the SubCtech data logger, evidenced by the distinct horizontal lines in figure 53 at 50 Hz and at harmonics (i.e. integer multiples of 50 Hz) above this frequency. The planned use of a battery power supply for the SubCtech data logger should alleviate this source of signal contamination.

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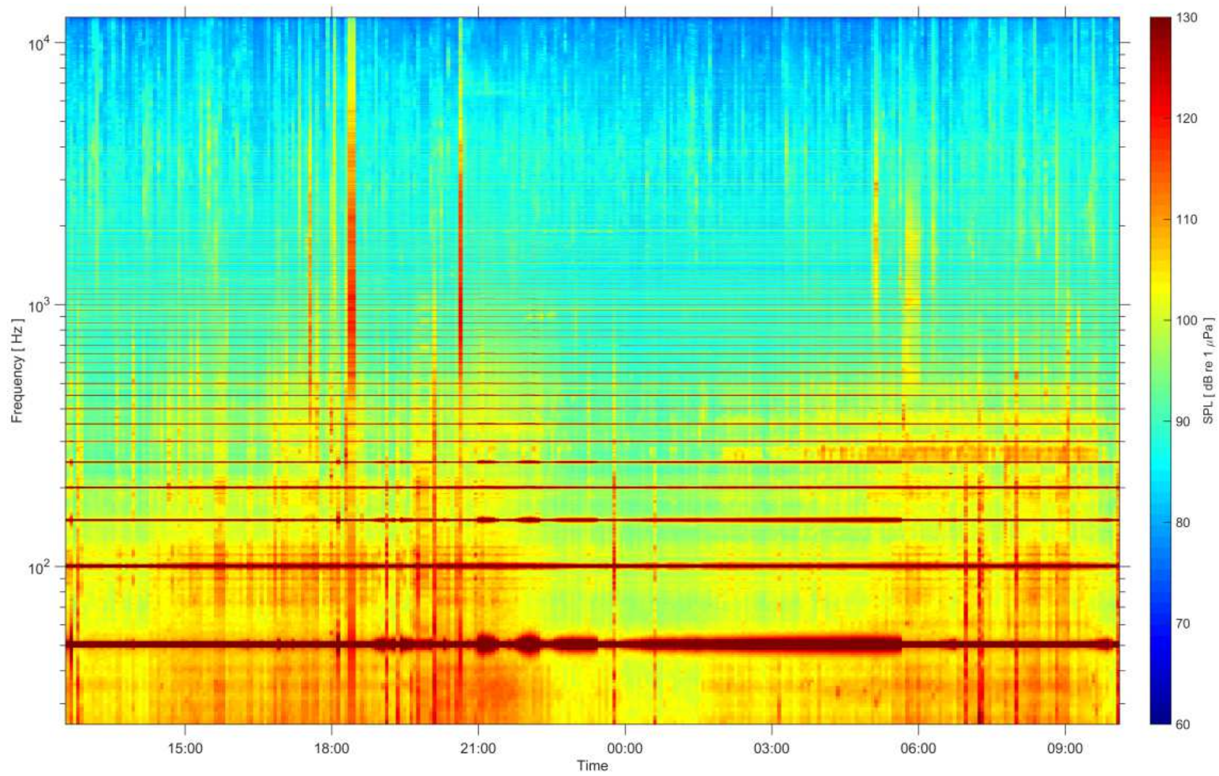

Fig. 53. Power spectral density of recorder deployment data from 28-29 September 2016. Data are at 1-Hz frequency resolution, presented as 60-s averages

The dynamic range of the noise recorder was well suited to the prevailing noise conditions, as shown by the spectral probability density of the data in figure 53. This demonstrates that the vast majority of data was recorded was within the dynamic range of the device, and that there was no apparent saturation of the signal. The noise floor of the instrument at 59 dB re 1 μPa^2 Hz^{-1} did not constrain the measurements.

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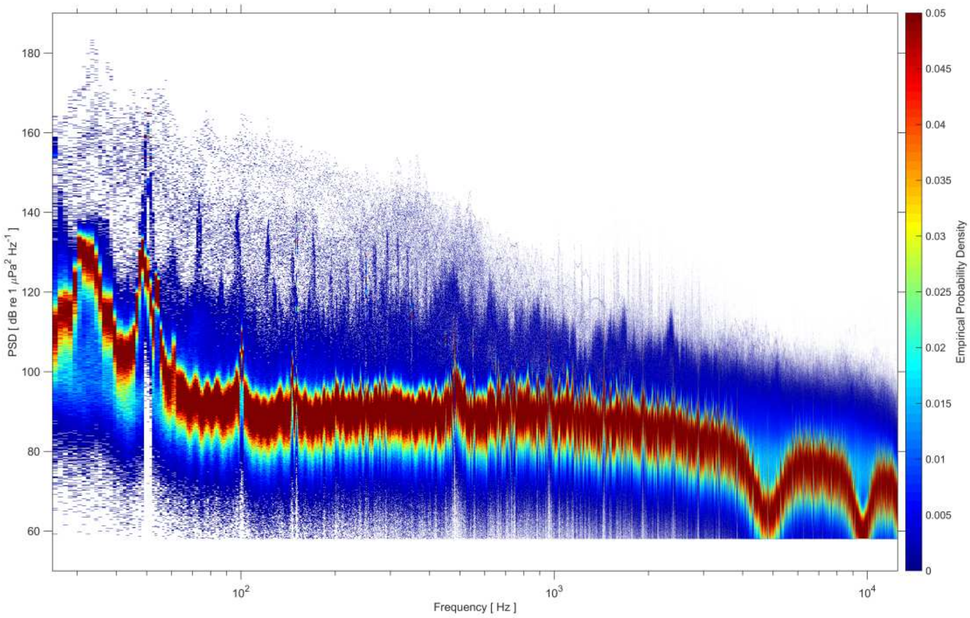

Fig. 54. Spectral probability density of recorder deployment data from 28-29 September 2016. Data are presented with 1-Hz frequency resolution.

The summary data at each of the octave bands indicates broad agreement with the raw data.

3.6.3 *Short description of stakeholders involved in testing activities with feedbacks;*

Four deployments have been conducted during the development phases of this project. The Institute of Oceanology Polish Academy of Sciences (IOPAN) are also involved in WP8, specifically for the delivery of Task 8.2.

IOPAN has been extremely helpful in assisting with the deployment of the noise recorder throughout the later stages of its development. During the third trial in the Baltic Sea, IOPAN deployed an off-the-shelf noise recorder alongside our noise recorder, and later during the trial in Oristano with Nathan Merchant (Cefas) providing the analysis. Both are described in 3.6.2.

During the tests in Oristano the sensor has been tested for several days onboard a small vessel in collaboration with the local Marine Protected Area "Sinis - Mal di Ventre".

3.6.4 *Possible optimization actions for prototypes design and performances after testing activities;*

The following future opportunities are beyond the scope of this project, but demonstrate the flexibility of the system design to be adapted for other target markets (see Annex 7 with updated highlights and technical description of the sensor than in previous reports):

User Software

Some of the data acquisition parameters require the pressure housing to be opened to make adjustment, such as the logging period and sampling frequency. Should the Noise Recorder be developed further, the facility to adjust these parameters via the user software could be included.

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Dual Hydrophone

A second hydrophone can be added to the system to enable the calculation of differential noise. This facility now exists within the packaging design so additional software running on the cRIO module would be required to implement this feature.

Automatic Gain Control (AGC)

The gain setting of the pre-amplifier is manually set by Cefas engineers prior to deployment. This setting is made based on experience from previous deployments. If, after reviewing the logged data from a deployment, a change of gain is required then again this would need to be set manually. The facility exists within the hardware of the CIPC module, to control the gain setting, additional control software would be required. A period of data analysis would be needed to determine and set the optimum gain level during a data acquisition cycle, and then to set it accordingly.

- ***Interface protocols***

A few minor communication protocol updates were recommended during the integration work with SubCtech, and following the trials in Oristano. These are only applicable if trying to interface the noise sensor to another common logger platform, so have not been incorporated here.

3.6.5 *Limits and optimal installations or possible improvements and definition of further possible optimization actions.*

Some of the final trials in Oristano included the use of mains power supplies to power the sensor, which introduces 50Hz noise to the system. This noise was not observed during the second deployment in the Baltic Sea, where the unit was battery powered. In normal operation the system will be deployed away from 50Hz mains noise sources.
Consideration to minimise external cable lengths will reduce electrical pick-up where the unit is not submerged in water.

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3.7 Final testing site with all partners and sensors: Oristano, 26th -30th September 2016

A final test involving all sensors has been organised at CNR in Oristano, five days long at the end of September 2016 (September 26th to 30th) and before the COMMON SENSE meeting (figure 55). Partners started the first day with a common test at CNR facilities in Torregrande (Oristano), where two tanks are available to be used for experiments: one tank able to be filled with 2300 l and a second one of 9000 l (see figure 56). In these tanks it was possible to have controlled parameters like water temperature, salinity or current speed and direction.

After these indoor tests, in the following four days sensors were mounted in the Mistras coastal lagoon. The location was chosen for tests longer than half day being continuously controlled and close to the CNR institute (3 km).

Fig. 55. The testing area in Oristano

Here, in agreement with local fishermen interested in some COMMON SENSE activities, sensors were installed at the limit of the lagoon with the Oristano Gulf (open sea), in a very dynamic environment at a maximum depth of 1,60 m and several facilities available like electricity (figure 57).

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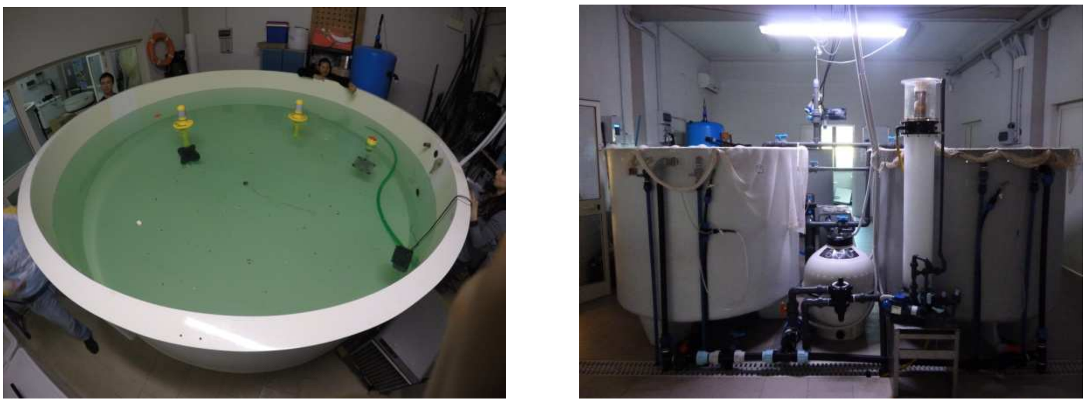

Fig. 56. The tanks available at IAMC CNR in Oristano for experiments: 2300 l (left) and 900 l (right)

Fig. 57. Aerial view of the testing area (left) and of the wharf that was used (right)

Fig. 58. Laboratory (left) and Mistras lagoon (right) tests

Sensors for the measurements of underwater noise, heavy metals, nutrients, microplastics and water temperature pH and pCO2, were initially tested at CNR facilities in Oristano where a 2300 litres tank was filled of sea water with controlled temperature, salinity and current speed, and other tanks were available in case of need. Here partners worked altogether in order also to connect and integrate their sensors to a data aggregator called SSU developed in the framework of the COMMONSENSE

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project and implement a near real time communication of data from the instruments to the SSU system and to WebPlatform

In the following days, also during the meeting, experiments went on with several sensors at Mistras lagoon apart underwater noise that was deployed in the touristic harbour of Torregrande continuously for several days to acquire the passage of boats and verify its limits as prototype. The location was chosen being a noisy environment more than the lagoon and thanks to the collaboration and interest of the local Marine Protected Area (MPA) "Penisola del Sinis - Isola Mal di Ventre" (http://www.areamarinasinis.it/). The MPA has left its facilities available to COMMON SENSE like a motorboat berthed at the touristic harbour close to the CNR institute, where the underwater noise sensor was tested.

On Thursday and Friday took place General Assembly, focused on remaining actions, both in technical and dissemination/ exploitation side. Short overview of technical development on lab six months, most of technical activities has been closed within this period, was showed on Thursday Morning. On Friday all tests were definitively concluded and sensors packed to be sent back to partners.

TABLES AND FIGURES CAPTIONS

TABLES

FIGURES

The COMMON SENSE project has received funding from the European Union's Seventh Framework Program (Ocean 2013-2) under the grant agreement no 614155.

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ANNEX 1 - SENSORS DESCRIPTIONS

The following Annex 1 contains all information gathered on sensors and their characteristics during the life of WP9, but mainly from March 2015 to September 2016, from sensors developers.

This file was particularly useful as it contains also suggestions on the possible platforms that could be used during testing activities.

It was continuously updated by sensors developers and let available in the COMMON SENSE area on Basecamp (https://launchpad.37signals.com/basecamp).

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ANNEX 2 - PLATFORMS DESCRIPTIONS

Similarly to Annex 1, also Annex 2 contains all information gathered on available platforms and their characteristics during the life of WP9 from sensors testers. After April 2015 this can be considered as the update of D9.1 on platform characteristics.

It was continuously updated by sensors testers and let available in the COMMON SENSE area on Basecamp (https://launchpad.37signals.com/basecamp).

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ANNEX 3 - SHORT SUMMARY OF TESTING ACTIVITIES

In Annex 3 tests are shortly described for each Work Package and, in brackets, its responsible partner. This is a resume of Excel tables continuously updated by partners and available in the COMMON SENSE area on Basecamp.

WP4 (CSIC)

Temperature & Pressure - WP4 (CSIC)
I testing – CSIC 2nd July 2015: laboratory temperature controlled test in sea water in Barcelona
II testing – CSIC/IOPAN 13th – 20th June 2016: temperature tested on Oceania cruise Gdańsk (Poland) - Tromsø (Norway)

pH - WP4 (DROPSENS)
I testing - DROPSENS/CNR 21st - 22nd March 2016: water sampled at Oristano and pH sensor tested at Sassari Univ. (stakeholder)
II testing - DROPSENS/CNR 21st - 22nd April 2016: water sampled in Oristano and analysed for pH at Sassari University (stakeholder)
III testing - DROPSENS/CNR 19th June- 04th July 2016: Arctic base in Ny-Alesund (Svalbard Archipelago, Norway). Water sampled during short cruises for laboratory analyses for pH.

pH & pCO2 - WP4 (CNR ICTP – FTM-UCM)
I testing FTM-UCIM/CNR 27th-28th October 2015: tested pH in simulated sea water FTM
II testing - CNR 21st - 22nd April 2016: water sampled in Oristano and analysed for pH at CNR laboratories in Naples
III testing FTM-UCIM/CNR 29th March and 20th-27th April 2016: tested pH in simulated sea water FTM
I testing FTM-UCIM/CNR 14th-16th June 2016: tested pCO_2 in sea water in Lucrino Gulf , Naples (I)

WP5 (DCU)

Eutrophication - WP5 (DCU)
I testing – DCU/TelLab 19th– 23rd December 2015: Floating Pontoon at the Hawai'i Institute of Marine Biology (Hawaii, USA)
II testing – DCU/CNR 25th November – 14th December 2015: CNR cruise in the Western Mediterranean
III testing – DCU/CNR 19th June – 4th July 2016: CNR Arctic Base in Svalbard Archipelago

WP6 (LEITAT)

Microplastics - WP6 (LEITAT)
I testing – IDRONAUT/CNR 25th November – 14th December 2015: CNR cruise in the Western Mediterranean
II testing - LEITAT /FNOB 14th May 2016: coastal navigation close to Barcelona

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III testing - LEITAT/IOPAN 13th – 20th June 2016: cruise Gdańsk (Poland) - Trømso (Norway)
IV testing - LEITAT /CNR 26th – 28th September 2016: Common deployment.
V testing - LEITAT /FNOB 6th November 2016: Vendée Globe yacht racing, including previous sensor installation and verification

WP7 (DROPSENS)

Heavy metals - WP7 (DROPSENS)
I testing - DCU/DROPSENS/CNR 25th November – 14th December 2015: CNR cruise in the Western Mediterranean
II testing – DCU/CNR 19th June – 4th July 2016: CNR Arctic Base in Svalbard Archipelago

WP8 (CEFAS)

Underwater noise - WP8 (CEFAS)
I testing - CEFAS/CEFAS 2-4th September 2015: first deployment of the prototype noise sensor in Lowestoft harbour
II testing - CEFAS/IOPAN 29th October – 10th November 2015: cruise in the Gdansk Bay

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ANNEX 4 – TESTING ON R/V OCEANIA

Testing of the new pyro- and piezoresistive polymeric temperature sensor onboard the s/y Oceania, from Gdańsk (Poland; Baltic Sea) to Tromsø (Norway; NE Atlantic, 14th to 20th June 2016)

Introduction

IOPAN kindly offered a unique opportunity to test new sensor prototypes developed within the EU-COMMON SENSE Project during the annual transit of their s/y OCEANIA (figure 1a) from Gdańsk in the Baltic sea to Tromsø in the NE Atlantic (figure 1b). Their offer included access to surface water continuously pumped to the onboard laboratory.

This water was also been used for other experiments carried out by the IOPAN scientific team. Among those there was a continuous measure of temperature and salinity by means of a freshly factory calibrated CTD SBE49 FastCAT. Testing a new temperature sensor in this framework was foreseen to have several advantages such as:

- Precision temperature contrast with a calibrated sensor
- High temperature variations
- High salinity variations
- Sensor in turbulent flow
- Possible stormy conditions

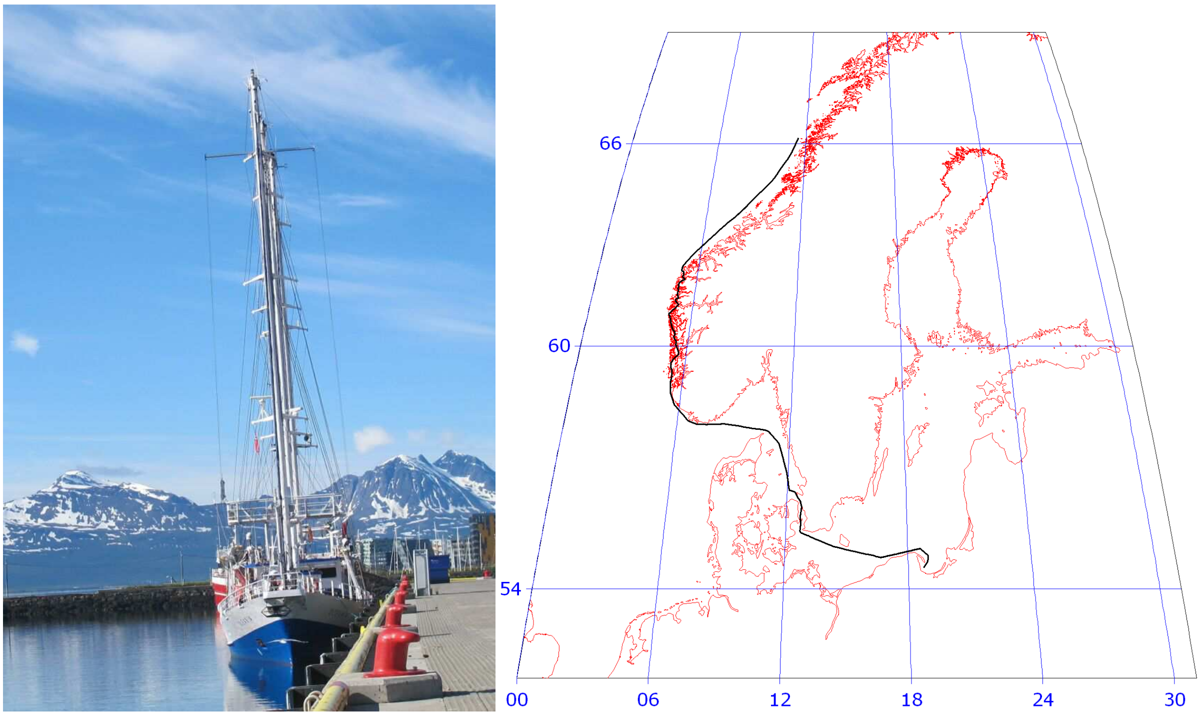

Fig. 1. S/Y Oceania in Tromsø harbour and her track, up to June 19 at 06:30 GMT

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Since a first prototype of the new pyro- and piezoresistive polymeric temperature sensor was successfully tested in air and fresh water, it was decided to take advantage of the transit offered by IOPAN and carry out a first sea test of the sensor in real sea water conditions.

Preparation

Set-up of the sensor

Three new prototypes of temperature sensor devices, able to be used in seawater, were prepared at the ICMAB-Nanomol facilities. The prototypes were designed to have a cooper housing in order to allow a good thermal conductivity and prevent a direct contact of seawater with the sensing element as well as with the contacts and wires. The resulting sensor device is shown in figure 2.

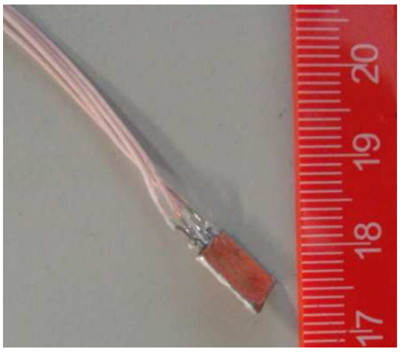

Fig. 2. Temperature sensing device. The sensing element is located inside the copper housing. Four wires coming out from the sensor for electrical measurements (scale is in cm)

All sensing devices were based on a temperature sensitive bi-layer (BL) films, acting as sensing elements, previously developed by the Nanomol group. The electrical contacts to the temperature sensitive BL film were positioned so that the resistance roughly varied from 30000 to 10000 Ω, for the typical range of seawater temperatures (-2 to 32°C). The contacts were made by evaporation of 10 nm thick layers of Cr, as adhesive layers, and 100 nm of Au using a nickel mask.

The sketch of the assembly showing the distance between contacts is shown in the figure 3. The resistance of the 3 sensing devices at room temperature was in the range of 24-27 kΩ.

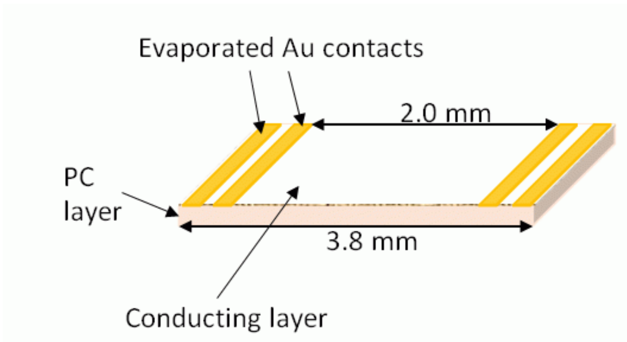

Fig. 3. Sketch of the four Au contacts, evaporated over a conducting surface of the BL film. Thickness of the evaporated contacts was 100 nm of Au plus 10 nm of adhesive layer of Cr. The width of the contacts was 150 nm

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Electrical contacts between the evaporated Au over the BL film and wires were made using a commercial silver paste, figure 4 (left). An insulating polycarbonate thin layer was applied over the contacts and wires to prevent shortcuts between the conducting surface of the sensing element, the Au contacts, the wires and the copper housing, figure 4 (right).

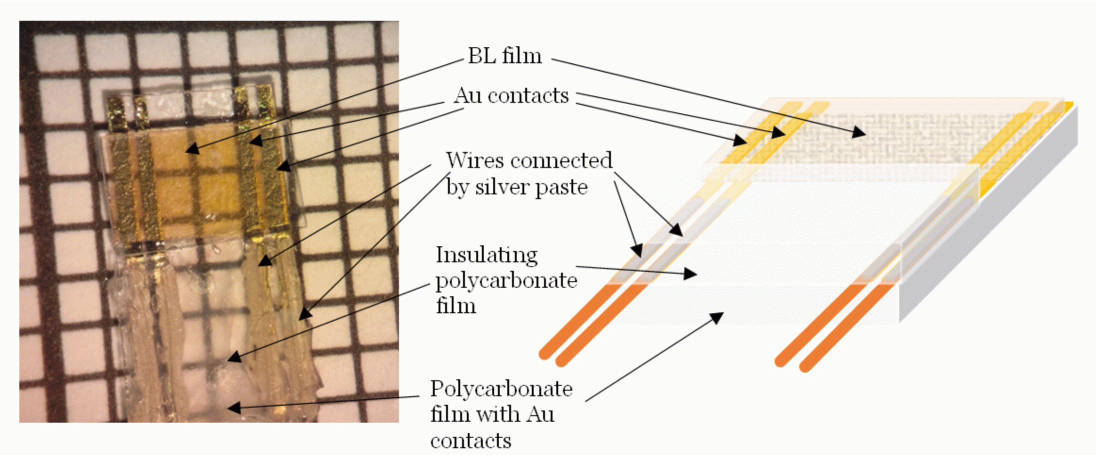

Fig. 4. Electrical contacts with BL film and its insulation from the housing: (left) photo image, (right) schematic view

Gold contacts with the same geometry as before were evaporated using the same nickel mask over a PC film. The BL film with the conducting layer down was placed over the latter polycarbonate (PC) film in such a way that the Au contacts of BL film make electrical contacts with Au contacts of the PC film (Fig. 3). Then both films were pressed and fixed using “SuperGlue”.
The free parts of the Au contacts of PC film were connected with the wires using a commercial silver paste. In order to prevent the shortcut between the wires and the copper housing, an insulating PC film was placed over the wires.

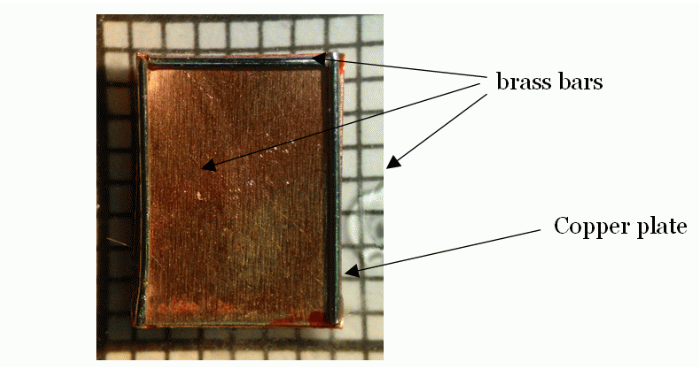

Fig. 5. Inside the housing of the sensor

The copper housing was made of 2 copper plates 100 µm thick with three 500 µm thick brass bars placed in the 3 sides of the plate to create an internal space for the sensor and wires (Fig. 5). The assembled sensing element was introduced inside the housing through the open (fourth) side. The

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thickness of the assembled sensing element was about 400-450 µm; so the 500 µm thick brass bars gave enough space to locate the sensing element inside of the housing.

The second copper plate was placed over the brass bars and it was hermetically sealed using a soldering iron and Pb. The sensing element with wires was introduced though the open part of housing and finally it was hermetically sealed with an epoxy resin resulting in the hermetically sealed housing shown in figure 1.

Since the voltages measured were quite low it was advisable not to have long wires before the PCB conditioner to avoid signal losses as much as possible.

The length of the wires was about 10 cm.

PCB adaptation

A PCB conditioner developed by Tyndall (figure 6a) was tested with the new sensors to measure the response of the system. First tests offered a very low signal to noise (figure 6b) that has been corrected by introducing a 100 nF capacitor at the sensor input (figure 6c).

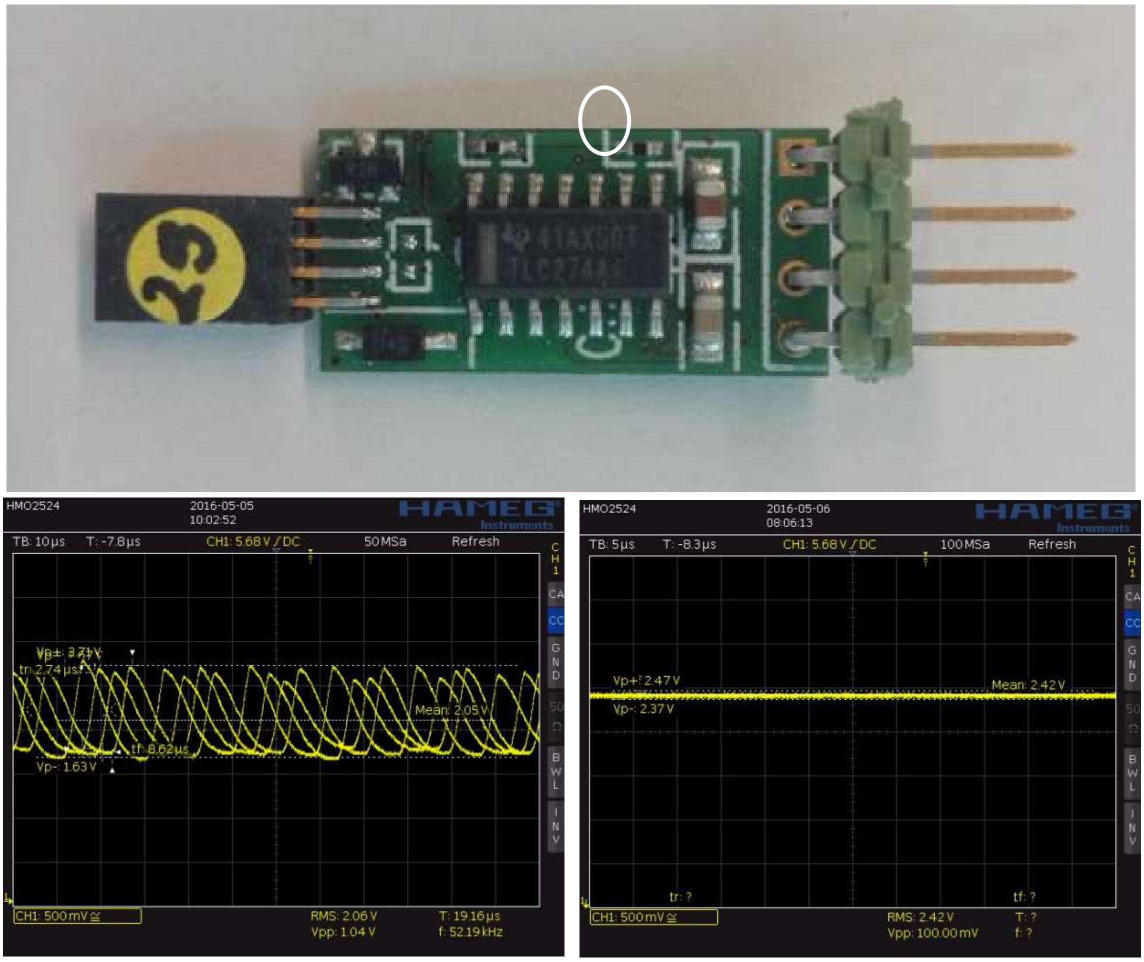

Fig 6. a) PCB conditioner (top), b) direct output from sensor+PCB shown by the oscilloscope (left) and c) the same output corrected with a 100nF capacitor (right)

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Since there was still no PCB for A/D conversion or any SSU adapted to this sensor signal, a provisional circuitry, adapted from that used in the ICM drifting buoys, was used instead.
The system was then adapted to 5V power supply as standard for USB connections. Since the original PCB conditioner was designed for a 12V power supply, according to the analog input requirements of SubCtech SSU (D4.3) a transformer DC/DC converter, 5 to 12V, was incorporated.
Due to the 12V design and the adaptations, the PCB conditioner (with the sensor) a higher consumption (175 mW) than really needed was detected. This may be an important issue if the system has to be battery powered for long time deployments.

A/D conversion and data transmission

The A/D converter used was a MPC 3424. It is a low noise-high accuracy 18 bit digital ΔΣ A/D converter from Microchip Technology Inc. These device can convert analog inputs to digital codes with a resolution up to 18 bit. (http://ww1.microchip.com/downloads/en/DeviceDoc /2088c.pdf). Its capacity was limited to a 2.048V so that the sensor+PCB response was adapted to this output, assuming that sensors were designed to vary their resistance from 30000 to 10000 Ω, as pointed out above. An additional reference voltage source of 2.00 V has been introduced to the board to check the A/D behaviour. A/D output was connected to an Arduino microprocessor that could be programmed from a computer through the USB connection (figure 7).
It was set-up to produce an ASCII data sequence as follows: record counter, time (in seconds), sensor output and reference voltage. To have a large number of measurements at high frequency acquisition rate was fixed to 10 s. The total consumption of all the system was 285 mW
All the electronics was placed in a methacrylate tube roughly sealed to avoid accidental water sparks (figure 8). On one side it was a 4-pin connector for the sensor and on the other side an USB connector.

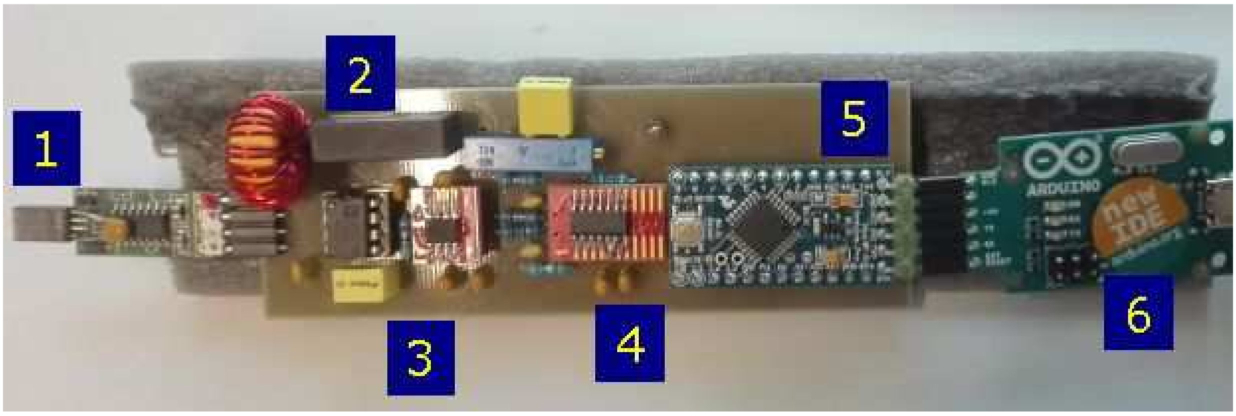

Fig. 7. Complete board used to connect the sensor and deliver a digital code through a USB. Tyndall PCB conditioner (1) to sensor including a 100 nF capacitor, DC/DC 5 to 12 V converter (2), reference voltage (3), MPC 3424 A/D converter (4), Arduino microprocessor (5) and USB serial converter (6) to computer

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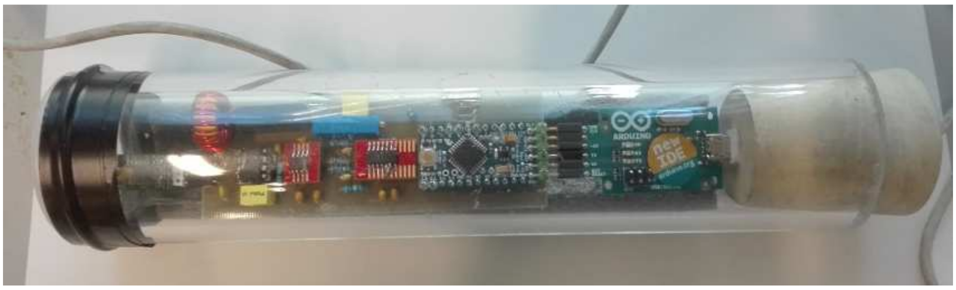

Fig. 8. Sensing electronics encapsulated in a methacrylate tube.

Laboratory testing

Before going to the ship every individual sensor has been tested in the laboratory. Sensor response was verified using different water mixture with ice and a Pt_{100} probe as reference. The results showed that:

- Sensor #1 performed well. Output was covering the whole range of temperatures.
- Sensor #2 failed probably due to water leaking inside the brass cover. It was discarded.
- Sensor #3 performed well but output voltage was out of range >2.048 V for temperatures lower than ~10°C.

Both sensor #1 and #3 were then ready for sea testing. It was decided to work with sensor#1 since it was expected to cover whole range of foreseen temperatures in the cruise. Sensor #3 would be used as a spare if the other had some failure.

On board

Set-up the system in the ship's laboratory

Since sensor assembly was quite delicate it was decided to put it into a 500 ml glass bottle (figure 9a). Three holes on the lid allowed for water input and output, and sensor cable to the electronics. Input water was connected to the source through a water distributor (Gardena®) allowing for 4 connections (figure 9b). The other three connexions were to serve the micro plastic sensor (see the corresponding report from Leitat), a three channel bbe Moldaenke fluorometer operated by IOPAN concurrently with micro plastic sensor and the remainder was used as overflow to control the rate through the used outputs (figure 10).

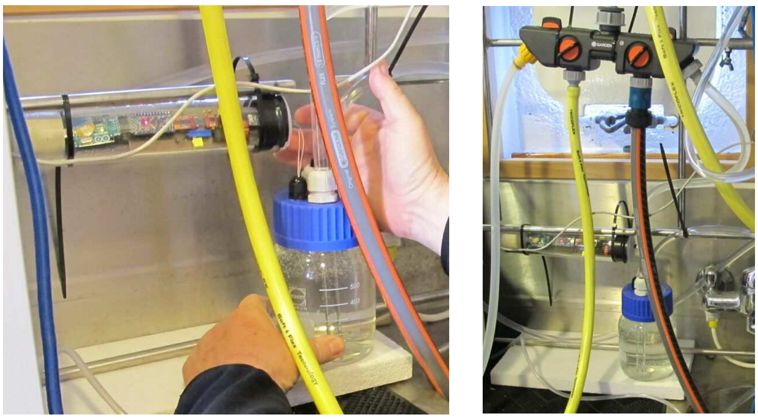

Fig. 9. New sensor installation at the Oceania laboratory

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Fig. 10. Instruments using surface pumped water underway at the Oceania wet laboratory

Flow rate was maintained between 1.5 to 2.5 l/min. It was enough to ensure a good water renewal within the glass bottle and not so high to produce excess turbulence that might affect the fragile sensor.

The USB output was connected to a PC and stored through capturing a hyper terminal (Table 1) application to a file. Files were closed every 2 to 8 hours and data was continuously displayed as to check the sensor behaviour.

12498;131084;1917343;2001328
12499;131094;1916343;2001312
12500;131105;1920171;2001328
12501;131115;1925375;2001312

Table 1. Partial reproduction of the sequences sent via hyper terminal: line #; time (seconds); sensor output (µV); reference (µV)

Data collection underway along the vessel's track

Part of the water pumped was diverted to the ship's deck where a SBE 49 FastCAT CTD was analysing temperature, conductivity and other variables required within the frame of the IOPAN experiments (figure 11). CTD data was recorded at 1s rate and geo-referenced by the ship's GPS. Other variables such as meteorological and navigation data were also recorded underway.

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Fig. 11. CTD on the Oceania deck measuring surface pumped water underway

All computers gathering information were synchronised with the GPS at GMT, as to have a unique time reference for all the data.
All the system was ready before starting the cruise at 22:00, 13 June 2016, at Gdańsk harbour.

1. Data from sensor

Sampling started using the above mentioned sensor#1, however a first look at the data stream revealed strong output noise. A verification of this output revealed a failure on the sensor that gave useless information (Table 2). It was probably due to water leaking inside the brass cover, as it happened with sensor#2 during laboratory tests, so that the sensor#1 was disconnected and replaced be sensor#3.

43;430;693062;2001328
44;440;833843;2001312
45;451;555906;2001312
46;461;513734;2001328
47;472;499734;2001312
48;482;474171;2001312
49;493;440562;2001312
50;503;428515;2001312
51;513;536687;2001312

Table 2. Sequence of data (as in Table 1) captured from sensor #1 starting the cruise.

After the connexion, just leaving the Gdańsk harbour at 00:00, 14 June 2016, sensor#3 did not produce any significant noise so that it was decided to run all the testing with this sensor while working (although data record from the first ~8h was lost because the computer automatically

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shifted to sleeping mode). In these conditions, it was assumed that the northernmost part of the transit will be out of range since temperatures beyond certain latitude were expected to be below 10°C.

Data provided by the sensor was reasonably stable and, indeed, beyond ~65°N most of the data was out of range as foreseen since water temperature was below 10°C (see figure 12)

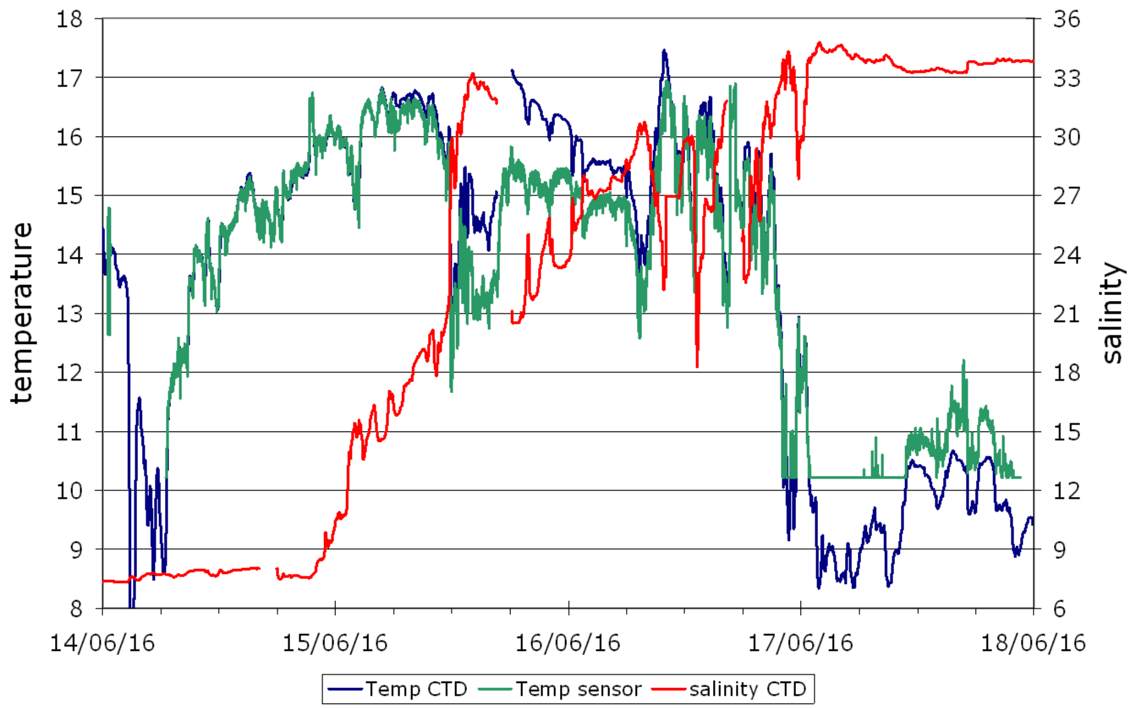

Fig. 12. Four days time-series of temperature from CTD and new sensor, and salinity from CTD underway the Oceania course up to 00:00 18/06

2. Data from CTD

No problem was found with CTD data. The instrument had been calibrated before the cruise and the only gaps in the sequence underway were due to few interruptions for maintenance of the IOPAN instruments on deck.

Data comparison and adjusting

Since temperature was linearly dependent on the sensor conductivity, an adjustment was performed to convert the reciprocal voltage to temperature, assuming a fixed current of 10µA, by comparing tipping points of the records such as maxima and minima. A first adjustment during the second day of sampling using 8 points (figure 13) within the temperature interval of 12 to 17°C in the Baltic sea. Linear adjustment was very good (r= 0.9990) and standard deviation of the residuals (0.0686°C) gave a reasonably good accuracy for the working conditions.

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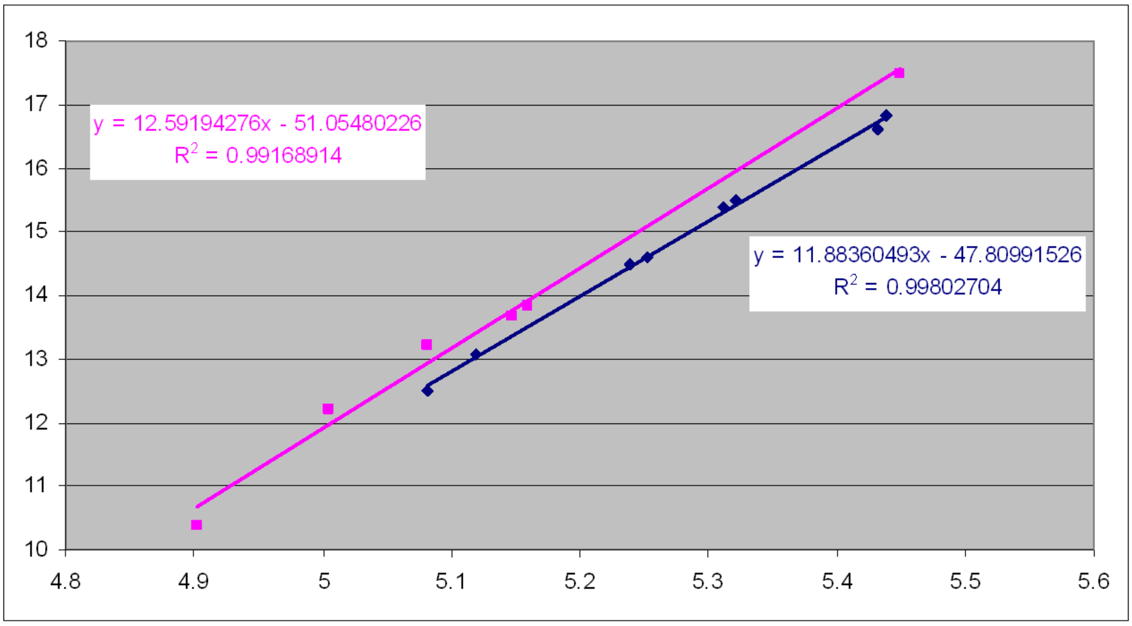

Fig. 13. Linear adjustment of sensor output converted to conductivity (µS) and CTD temperature. Blue dots (rightmost line) correspond to the first period (roughly before noon 15 June) and pink dots (left) to the second period (see fig. 12)

However after crossing the Danish strait, a significant deviation was found from sensor converted data and the CTD. A new series of 6 tipping points were used to find a new relationship giving a significant drift with respect to the previous one (ΔT ~ 0.5°C) and lower accuracy from residuals with double SD (0.1339°C).

This behaviour was quite surprising. No evidence of what might be the cause of the drift and lower accuracy. The only environmental condition that significantly changed was salinity, from less than 10 (Baltic) to more than 30 (North Sea). However it is hard to assume that a well sealed (otherwise it would not be working) sensor could be influenced by a change in water conductivity, even though it was a quite large shift. Any other conditions that could come from changes in deck conditions for the CTD measurements, such as environmental temperature or occasional sun exposition (really scarce all the time) seem not to be significant enough over the large flow rate (>10 l/min) flowing across the deck system. Of course, laboratory environmental conditions were almost constant all the time.

In addition, no changes in weather and sea conditions were found (low wind and waves) before entering later (17/06 early morning) in a heavy storm that apparently did not affect the sensor activity.

Results

Figure 12 shows a time series of CTD temperature and salinity, and sensor data converted to temperature using the first two-day adjustment. Note that the sequences are almost coincident at the beginning and shifted after crossing the Danish straits (~ at noon 15/06), as evidenced by the salinity record. It can also be noted that at the end of the time series, sensor readings are out of scale

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but they are recovering at points where temperature raised above the threshold but showing a different shift.

The whole sensor sequence has then three parts:

1. Mainly within the Baltic sea with high accuracy and very good adjustment.
2. After crossing the Danish straits with less accuracy and a negative shift (Sensor < CTD)
3. At the end of the series when temperature is generally below the sensor threshold showing a positive shift, where sensor temperatures are slightly higher than CTD.

Final test after cruise

The above results on the sensor#3 behaviour suggested that it might be suffering a time-dependent drift or may be affected by other environmental conditions such as salinity, housing corrosion, etc, as indicated above. In view of that, a complete laboratory test using sea water is planned after the cruise. The idea was to use the testing system already used in the first laboratory tests almost one year ago (see the report May 2015)

Unfortunately when testing in the laboratory in Barcelona was ready and sensor connected, a high noise appeared in the sensor output (figure 14) as it happened with the two previous prototypes. Therefore no test after the cruise could be done and the causes of the observed drift will remain unclear

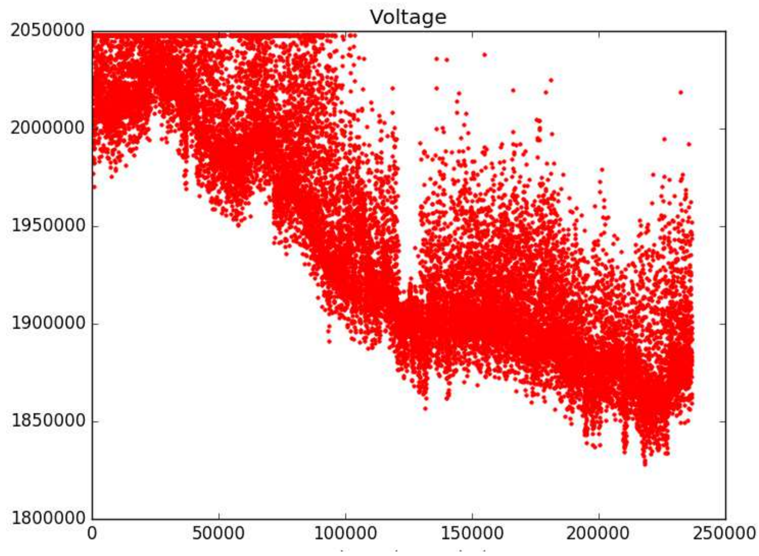

Fig. 14. Time series (sec) of sensor output (µV) in an attempt to reproduce a progressive temperature increase in laboratory conditions after the cruise

Conclusions

This is a very first test in real sea conditions of the new pyro- and piezoresistive polymeric temperature sensor developed by Nanomol-CSIC within the COMMON SENSE Project. Globally this first test can be assumed as very useful to show the pros and cons of the sensor behaviour, test the electronics and have an idea of what has to be done to integrate the sensor in any other device. Unfortunately a final test to understand the observed changes in response has not been possible.

The following points are intended to summarize of this experience:

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1. Sensor development:
   All the process to prepare the sensor has to be clearly improved. Especially contacts, housing and sealing against water leaking.
   All the ensemble is clearly too fragile against stresses on connexions and cables. It must be improved.
   Sensor resistance ranges should always be kept between 10000 and 30000 Ω for the typical seawater temperatures (-2 to 32°C).
2. PCB conditioner was working correctly but if modified for 5V instead of 12V DC power supply, consumption must likely be reduced.
3. A/D and processor. This is supposed to be developed within the SSU. All the present experience has been done using a board adapted from another use. It has been working properly but it is not the one developed within the COMMON SENSE consortium.
4. The whole system has shown good linear response, quite good accuracy but some uncontrolled shifts had been observed whose origin must be carefully studied in the laboratory.

Next steps will be to fix the sensor in a buoy and leave it for a while. To do this new step all the previous steps must be satisfactory solved.

Acknowledgements

We are very grateful for the invitation to participate in the cruise. This successful experience has been possible thanks to the collaboration with Sławomir Sagan and his scientific team from IOPAN. Our acknowledgment to the Captain and crew of the s/y Oceania that gave us all kind of help and facilities for our work and enjoy the trip despite some days of stormy weather.

ANNEX 5 – PH TESTING TECHNICAL REPORT

Analysis of the data collected during field pH testing activities in Oristano lagoons and the Arctic base

Oristano lagoons samples:

Samples from Oristano lagoons where collected during the months of March and April. The average temperature of the samples was +20° C with a salinity that varies from 12.28 to 38.5 PSU.

As described in paragraph above, once the electrode has been inserted in the sample, the potential was recorded after 120s.

Table 1 below shows the values of potential (mean of the last 3 measurements) obtained with the antimony sensor, the values of pH obtained with the laboratory pH-meter, and the salinity and temperature of the samples collected.

Date	Sample name	E mean (mV)	Ph lab	Salinity (PSU)	Temp (°C)
29-mar-16	Cabras 1	-470	8,68	13,07	15,64
	Cabras 2	-469	8,57	12,28	15,43
	Mistras	-413	8,04	36,66	15,09
	Scolmatore	-425	8,38	28,7	
01-abr-16	SG1	-427	8,05	30,84	20,4
SPE 1	SG2	-426	8,13	30,98	20,3
	SG3	-427	8,13	30,21	20,3
	SG5	-426	8,31	30,48	20,3
	SG7	-414	8,25	36,16	20,1
SPE 2	SG1	-426	8,05	30,84	20,4
	SG2	-429	8,13	30,98	20,3
	SG3	-428	8,13	30,21	20,3
	SG5	-428	8,31	30,48	20,3
	SG7	-417	8,25	36,16	20,1
18-abr-16	SMR	-425	8,12	25,4	22,2
	MIS	-418	8,19	37,7	22,2
22-abr-16	pontile	-418	8,16	38,37	20,7
	tortoli 3	-416	8,3	28,52	20
	is aruttas	-422	8	38,5	20,5
	scolmatore	-433	8,02	35,8	21,1
	tortoli 4	-414	8,03	34,42	20,9
	mistras	-421	7,84	38,25	21,8
	sa mardini	-428	8,1	26,88	21,3

Table 1. Values of potential, pH, salinity and temperature for the Oristano lagoons samples

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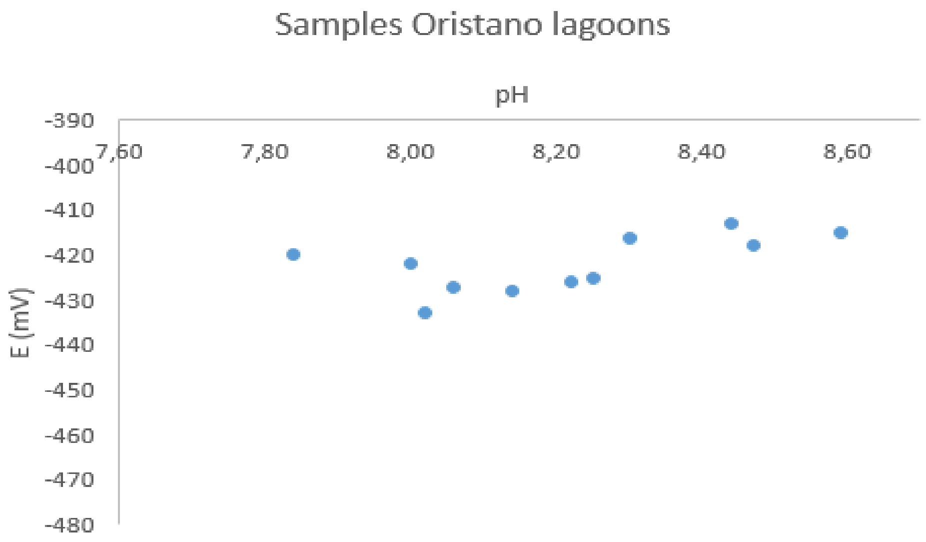

Fig. 1. Potential mean vs. pH values

The sensor showed reproducibility between potential measurements, with %RSD < 0.6% between different samples with same pH value and with a precision of 0.5 pH units.

Arctic base samples

Samples from Arctic base where collected on the 22nd of June 2016 during 7:15 am to 12:54 pm. The average temperature of the samples was +3.3 °C with a salinity that varies from 29.2 to 35.0 PSU. Also, the conductivity of the samples was measured with a mean value of 31.17 mS/cm. Maximum depth of samples collected was 300 m.

Table 1 below shows the values of potential obtained with the antimony sensor, the values of pH obtained with the laboratory pH-meter, and the salinity and temperature of the samples collected.

Station	hrs (UTC)	Long °E	Lat °N	Depth (m)	E mean (mV)	pH	Salinity(PSU)	Temp(°C)
ITA 001	7:15	11,980	78,940	0,0	-413	8,849	32,415	7,051
				150,0	-406	9,805	34,973	3,246
				298,0	-405	9,827	34,918	1,805
ITA 002	8:25	12,116	78,919	0,0	-414	8,957	33,520	4,660
				65,0	-417	9,26	34,932	3,864
				125,0	-409	9,433	34,975	3,228
ITA 003	8:43	12,167	78,913	0,0	-415	8,925	34,251	4,704
				55,0	-417	9,214	34,910	3,947
				105,0	-408	9,387	34,923	3,234
ITA 004	9:28	12,240	78,914	0,0	-409	8,946	33,964	4,219
				50,0	-409	9,241	34,878	3,767
				100,0	-404	9,444	34,923	3,183
ITA 005	9:51	12,262	78,903	0,0	-409	8,854	34,192	4,428
				35,0	-409	9,214	34,850	4,232

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Station	hrs (UTC)	Long °E	Lat °N	Depth (m)	E mean (mV)	pH	Salinity(PSU)	Temp(°C)
ITA 006	10:18	12,362	78,891	0,0	-407	8,844	33,242	3,134
				30,0	-409	9,133	34,674	4,114
ITA 007	10:56	12,465	78,874	0,0	-412	8,756	33,427	3,330
				25,0	-418	9,067	34,672	4,115
ITA 008	11:07	12,515	78,873	0,0	-412	8,947	33,130	2,434
				35,0	-414	9,256	34,756	3,223
				65,0	-410	9,403	0,000	0,000
ITA 009	11:53	12,490	78,892	0,0	-408	8,858	32,740	2,482
				35,0	-411	9,263	34,729	2,592
				70,0	-407	9,353	34,838	2,402
ITA 009 BIS	12:17	12,581	78,909	0,0	-403	8,846	29,184	3,474
				50,0	-403	9,524	34,668	0,185
				100,0	-401	9,771	34,674	0,011
ITA 010	12:54	12,439	78,905	0,0	-409	8,897	31,490	2,827

Table 2. Values of potential, pH, salinity and temperature for the Arctic base samples

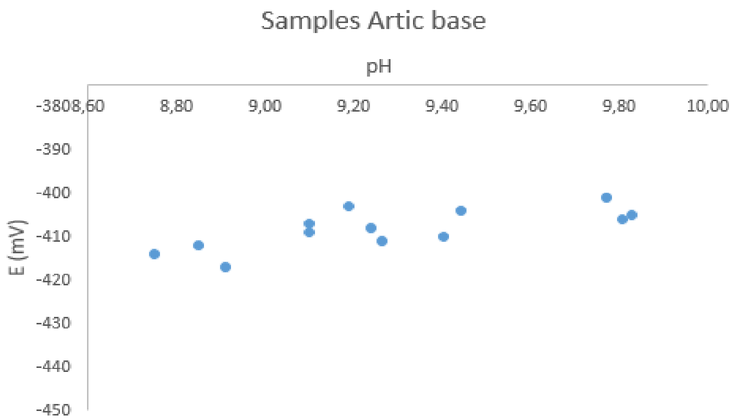

Fig. 2. Potential mean vs. pH values

When analysing data from Artic samples, the sensor showed reproducibility between potential measurements, with %RSD < 1.4 % between different samples with same pH value and with a precision of 0.5 pH units. With all the data obtained during field testing activities it has been concluded that:

- Antimony pH sensor showed a good response in the determination of pH in the range 7.84 – 9.827, in samples with salinity values between 12.28 and 38.5 PSU.
- The sensor is temperature sensitive, but results obtained with samples collected at +20 °C and +3.3°C can be compared with good response between different electrodes.

Precision of the samples has been showed to be 0.5 pH units. This value is not acceptable when measuring seawater samples, so the sensor needs to be optimized.

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ANNEX 6 – MICROPLASTIC PRE-TESTING REPORT

Here we report the results of the pre-deployment validation of the optical camera and optomechanical system before deployment, to detect problems that may occur in the deployment. Information about microplastics validation, can be found on D6.3 where information about individual performance was exposed.

Information regarding microplastics sensor can be found on:

- ✓ D6.1: Microplastics sensor technical specifications (M21)
- ✓ D6.2: Testing methodology definition (M28).
- ✓ D6.3 Proof-of-concept functional blocks and laboratory tests report (M30)

- ✓ D3.3 Sensor Data Management Module, including Sensor Data Management Module Specification (M24)
- ✓ D4.3 Integrated sensor arrays. Laboratory (M24)

As a summary, the following was tested:

- Communication with camera, in order to acquire images
- Communication with peristaltic pump
- Algorithm for microplastics detection
- Algorithm for microplastics quantification
- Communications with other components

Scenario description

After integration of the complete system, we performed the same validation tests described in D6.2, adapted to the fact that the optical block is now integrated, in order to test that, once integrated, the optical block still works according to specifications.

Specifically, we tested the following:

- ✓ LED operation
- ✓ Camera Operation & data capture
- ✓ Camera settings
- ✓ Focus
- ✓ Illumination homogeneity
- ✓ Dynamical measurement

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Results
LED operation
Procedure: Visual inspection that all LEDs emit and that their emission varies when turning the power knob.
Results: Pass
Initially, although all LEDs switched on, and changed the emission level when turning the power knob, after a certain voltage was applied, one of the LEDs started switching on and off continuously. After examining the components, we determined that one of the LED pins was bend too close to the other pin, inducing a small periodic electrical discharge once a threshold voltage was reached. Once this faulty LED was changed, the test was repeated with positive results.

Camera Operation & data capture
Procedure: With the device switched on, the software must be able to acquire images continuously.
Results: Pass
Once the system is powered, the camera acquires images satisfactorily.

Camera settings
Procedure: With the device switched on, the camera settings established by default by the user controller software must be adequate to capture the features of the image.
Results: Pass
With water flowing through the cuvette and the illumination switched on, the proper camera settings were found to allow the capture of clear, non-saturated images. The software henceforth establishes these settings whenever the microplastic analyser is switched on.

Focus
Procedure: Check that, with the illumination switched on, and with water with microplastic pieces flowing through the cuvette, the acquired images of the microplastic pieces are clearly defined and in focus.
Results: Pass
Once the optical system integrated in the microplastic analyser device, and with the water and microplastics flowing through the cuvette, the focus knob was adjusted so that the microplastic pieces (of different sizes) were well-defined within the image.

Illumination homogeneity.
Procedure: Check that the illumination on the plastic pieces flowing though the cuvette is uniform.
Results: Partial Fail → solved by SW analysis
It was found that when the illumination is switched on at the proper operation levels, there is a stray light reflection in the side of the cuvette, which made the software make the mistake of detecting an object were there was none (see the following Figure).

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Fig. 5. Wrongly identified object due to stray reflection on the cuvette surface

This stray reflection is due to the combination of cuvette geometry and a slight non-uniformity in the illumination. For now the detection glitch has been solved via software, and we are currently working in modifying the illumination source in order to eliminate the stray reflection.

Measuring while sampling dynamically.

Procedure: Check that, with water flowing through the cuvette at the normal speed of operation, and with the illumination and camera settings established at their proper values when initializing the device, the outline of the plastic pieces in the acquired images is clearly defined.

Results: Pass

The following figure shows image of several microplastic pieces flowing through the cuvette at the normal speed of operation.

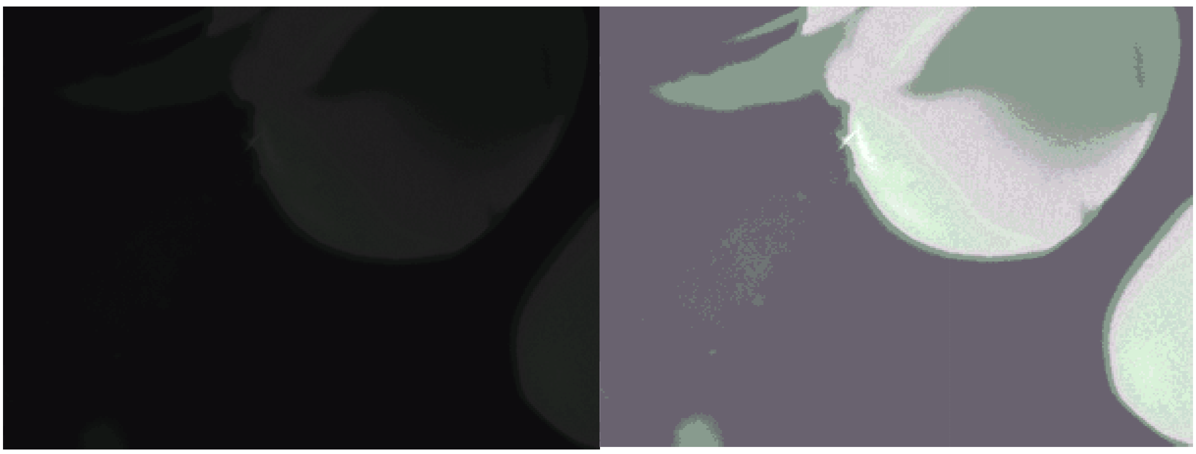

Fig. 6. Several pieces of microplastic flowing through the cuvette (left original, right high contrast picture)

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The image is sufficiently clear for the software to distinguish adequately between the different pieces of plastics, and also give an adequate estimate of their dimensions.

Data and images storage.

Procedure: Check that, the USB memory disk is mounted correctly in the initial state, the application automatically start running, force the Micro plastics go through the corvette and observe the data changes in the result file, check the image storage in the USB memory disk.

Results: Pass

The following figure shows when there is no Micro Plastic pass through the cuvette, the last column of the results display all 0,000; however when detected microplastic, the last column display the non-zero data, meanwhile the captured image is saved in the USB memory.

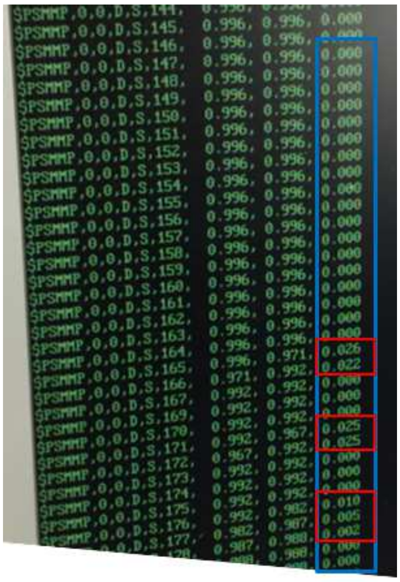

Fig. 4. Processing results with detected microplatic

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ANNEX 7 – UNDERWATER NOISE TECHNICAL SUMMARY

Key information:

Highlights	Technical Description
Utilises cost effective state of the art technology, with core components sourced from well-established suppliers ensure good ongoing support. Design is based upon a modular format and provides Open Source operating code to provide greater flexibility and adaptability. Differential hydrophone output minimises the effects of electrical pick up. In-field check of the hydrophone performance using a piston phone with custom adaptor. Fully interfaced with the SSU to enable integration with other sensor data. Raw data is logged to internal memory; summary data is derived for transmission to provide live data. Hardware / software interface enables further development for use in other deployment applications, and stand-alone operation. Custom Acetal housing provides good protection from environmental conditions.	Records noise data in the frequency band 10Hz to 10kHz. Adjustable sampling frequency 1.6kHz to 50kHz. Sensitivity to provide 100dB dynamic range that can be configured between 59dB to 180dB ref 1µPa. On-board processing algorithm to provide Power Spectral Density summary data that can be transmitted to the SSU. This includes one-third octave bands at centre frequency 63Hz and 125Hz. 512GB SSD Memory (expandable) Software interface to assist with pre-deployment tests. Power Supply 24v @ 1.87W. Weight 9.3kG (in air). Size : Height 541mm Diameter 195mm. 

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