

Joint European Research Infrastructure network for Coastal Observatories



Second assessment of FCT activities D#1.8

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1. Document description

REFERENCES

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2. Introduction

Context

The Forum for Coastal Technology is part of the Jerico project. It aims to promote greater interaction between the scientific requirements and related market for marine sensors and equipment, including better feedback from users to makers on improved design for ease of use. Ultimately it should help to bring closer the industry and the research communities with the objective to allow a substantial progression of research in public and academic institutes to products and services. The Terms of Reference (www.jerico-fp7.eu/coastal-technologies) describe in details the aims as well as the main content and strategic issues raised by the FCT.

The first FCT workshop conclusion

The first FCT Jerico workshop was organized in October 2012 at the event 'Sea Tech Week' (France). It gave the opportunity for both scientists and company representatives to discuss needs from the field of in-situ sampling and monitoring. With a broad scope, the workshop focused on dissolved oxygen (DO) and nutrients.

A couple of online surveys were carried out towards both scientists and the sensor companies. They emphasised the requirement for better sampling dissolved oxygen and nutrients. The results from these surveys are available in the report published after the first workshop. (<http://www.jerico-fp7.eu/coastal-technologies>)

Following this first workshop, a white paper on DO was written (Laurent Coppola *et al.*). It outlines the key role that DO plays in biogeochemical cycles, but also highlights the specific scientific needs (accuracy, reliability, robustness) as well as the substantial difficulties to calibrate the sensors (mainly optodes in green water).

Indeed, DO sensor calibration and maintenance is time consuming and a costly operation that is mostly only affordable by large public research institutes. Thus scientists often calibrate their sensors for their own applications. However, these scientists as well as companies and other users (coastal environmental activities for regulation or management) lack information about the best calibration methodologies and practices. This was confirmed by a DO inter-calibration experiment carried out (beside the workshop) in Ifremer's specialised laboratory and in a CNRS laboratory (Observatoire Océanographique de Villefranche/Mer).

Finally all attendees agreed that a dedicated 'calibration workshop' would give opportunity for a better exchange about 'know-how' between manufacturers and sensors users. Strong interaction with the industry is necessary to maintain sensors at a good level of performance.

Here after are reported results after DO intercalibration experiment and a synthesis of discussions led in the 2nd calibration workshop.



3. DO sensors in-situ inter-comparison exercises

3.1 Material and Method

In the context of the JERICO Work Package 10, different Dissolved Oxygen (DO) sensors have been inter-compared at the Observatoire Océanologique de Villefranche-sur-mer (OOV, CNRS, France).

Two field experiments were realized in the Villefranche Bay, one in October 2013 and one in December 2013, using several types of DO sensors.

- Classic sensor (electrode membrane):
 - o 1 SBE 43 (OOV-CNRS, France).
- Optical sensors:
 - o 1 optode SBE 63 (M.I.O./CNRS, France),
 - o 1optodeAanderaa 3975 (HCMR, Crete),
 - o 1 optode Aanderaa 4330 oxygen optode (OOV-CNRS, France),
 - o 1 HOBO U26 ONSET (MI, Ireland)

Each time, a vertical DO profile has been associated with a CTD profile (SBE 19+) with water sampling for Winkler titration (used as O₂ concentration reference).

15-Oct-2013

Just before the JERICO WP10 meeting at Villefranche-sur-mer, Laurent Coppola (OOV), Emilie Diamond (OOV), Ingrid Puillat (IFREMER) and Manolis Ntoumas (HCMR) performed a sea experiment for testing accuracy and precision of existing DO sensors but using different technologies and calibration procedures. For this experiment, we used a SBE43 and a SBE 63 (connected directly on the SBE 19+) and two different Aanderaa optodes (autonomous).

The first cast was realized with the SBE 19+/ SBE 43/SBE 63 unit(Figure 1)near the EOL buoy in the Villefranche Bay with a 30min stage at 25m. Then seawater was sampled at 25m depth via a Niskin bottle to get three Winkler samples.

Two other casts were performed at 6 m depth with the same unit and using firstly an optode 3975(Figure 2) and secondly an optode 4330 (Figure 3). These optodes were directly connected to a PC on board. Three other Winkler samples have been taken from a Niskin bottle at 6 m.

The water samples were analyzed later with the Winkler method, using a Metrohm 702 SM Titrino potentiometric titration.



Figure 1:Autonomous CTD/DO used for JERICO exercise (SBE 19+/ SBE 43/ SBE 63)

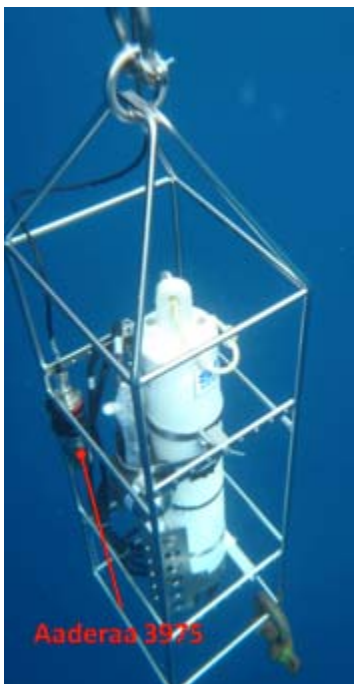
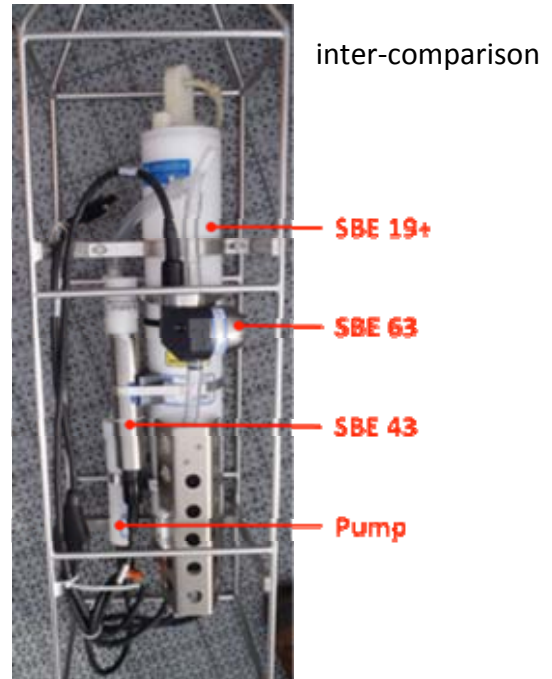


Figure 2: CTD/DO used for JERICO inter-comparison exercise (SBE 19+/ SBE 43/ SBE 63/ Aanderaa 3975)

Figure 3: CTD/DO used for JERICO inter-comparison exercise (SBE 19+/ SBE 43/ SBE 63/ Aanderaa 4330)





22-Oct-2013

A second experiment has been performed later in October 2013 at the same location (Villefranche bay). On this cast, we deployed a SBE63 sensor on the SBE19+ and we mounted on parallel the HOBO sensor working autonomously. The cast was deployed at 50m depth for 30 min inter-comparison exercise. We collected as well 3 bottles for Winkler measurements.

16-Dec-2013

Another cast was realized near the EOL buoy in the Villefranche Bay, but this time with a 20min stage at 50m and added to the SBE 19+/ SBE 43/ SBE 63 unit, the autonomous HOBO U26 (Figure 4) Then sea water was sampled at 50m via a Niskin bottle to get three Winkler samples analysed two days later.



Figure 4: Autonomous CTD/DO used for JERICO inter-comparison exercise (SBE 19+/ SBE 43/ SBE 63/ HOBO U26)



3.2 Results

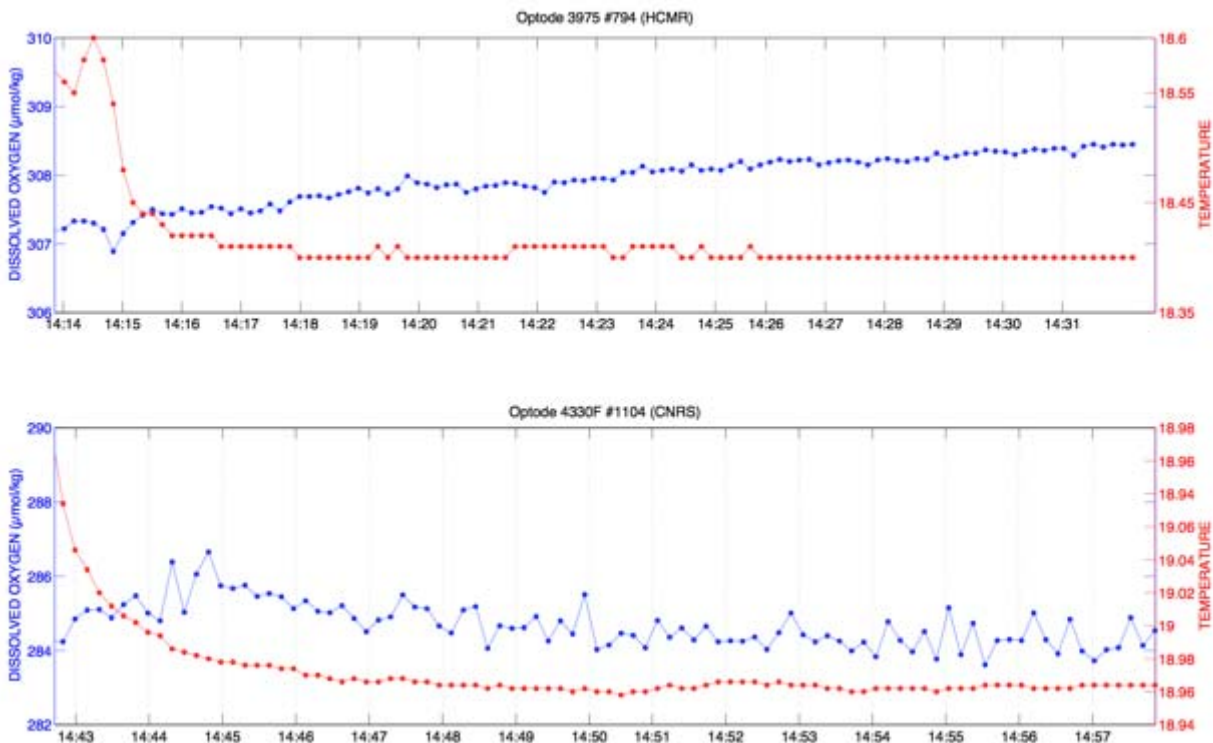


Figure 5: Oxygen results for the 2 optodes deployed in surface waters in October 15th. The Winkler measurements at this depth ranged around 245-250 µmol/kg.

The two optodes (3975 and 4330) showed different results during the deployment. First of all, in between the two optodes, we observed an offset of 20 µmol/kg. The different performance between the two optodes might explain this discordance. For example, the new foil installed on optode 4330 provides a faster time response than the optode 3975 (Figure 5). This could explain the larger variability of O₂ concentrations observed on the optode 4330 data. Secondly, the new multi-point calibration procedure performed by the manufacturer improved the quality and the accuracy of the optode 4330 measurements. Compare to the O₂ reference value (Winkler titration indicated an oxygen concentration around 245-250 µmol/kg), we observed a large offset ranging from 35 to 55 µmol/kg for optode 4330 and optode 3975, respectively. Even if the results from the optode 4330 is better than those from optode 3975, this offset is too large to be due to natural variability. Such offset for both optodes could be explained by a too old calibration procedures and a long storage of sensors to the dry air condition.

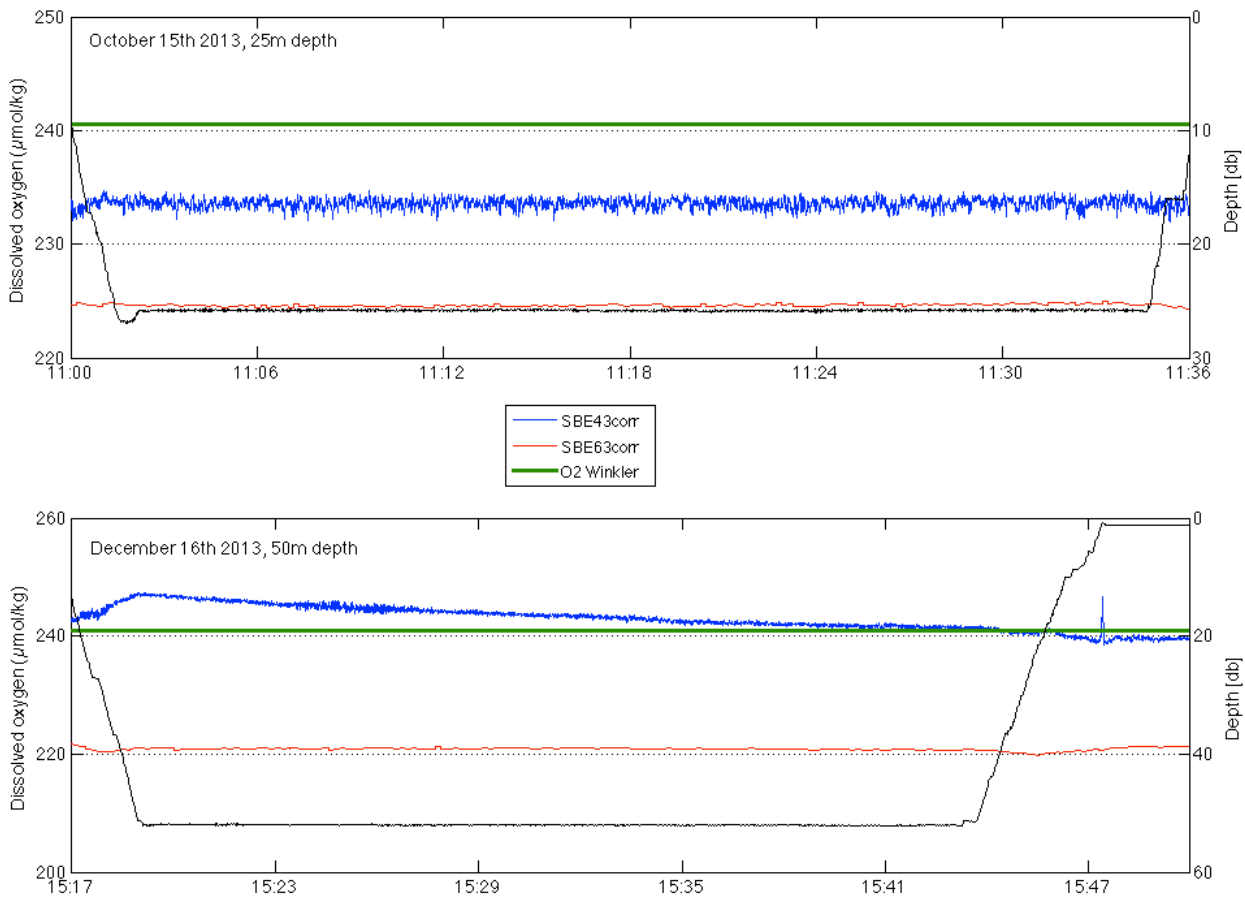


Figure 6: Results for SBE43 and SBE63 sensors deployed at 25 and 50m depth in the Villefranche-sur-Mer bay. The green line represents the Winkler reference.

Results from the two SBE sensors indicate also a disagreement. However, the SBE43 data after correction (adjustment of the SOC slope coefficient) show a better response compared to the SBE63 (adjustment of T, P and S) for the two deployments (Figure 6). The recent yearly calibration of the SBE43 could explain this difference. On the other hand, the SBE63, which corresponds to an optode, is very sensitive to the dry air temperature and the long storage before the deployment. This might explain the observed offset between SBE63 data and the O₂ Winkler reference value (20 µmol/kg). However, the capacity of the optode to not drift over the long deployment could give an advantage to this sensor compared to the SBE43 very sensitive to the biofouling and the cleaning procedure.

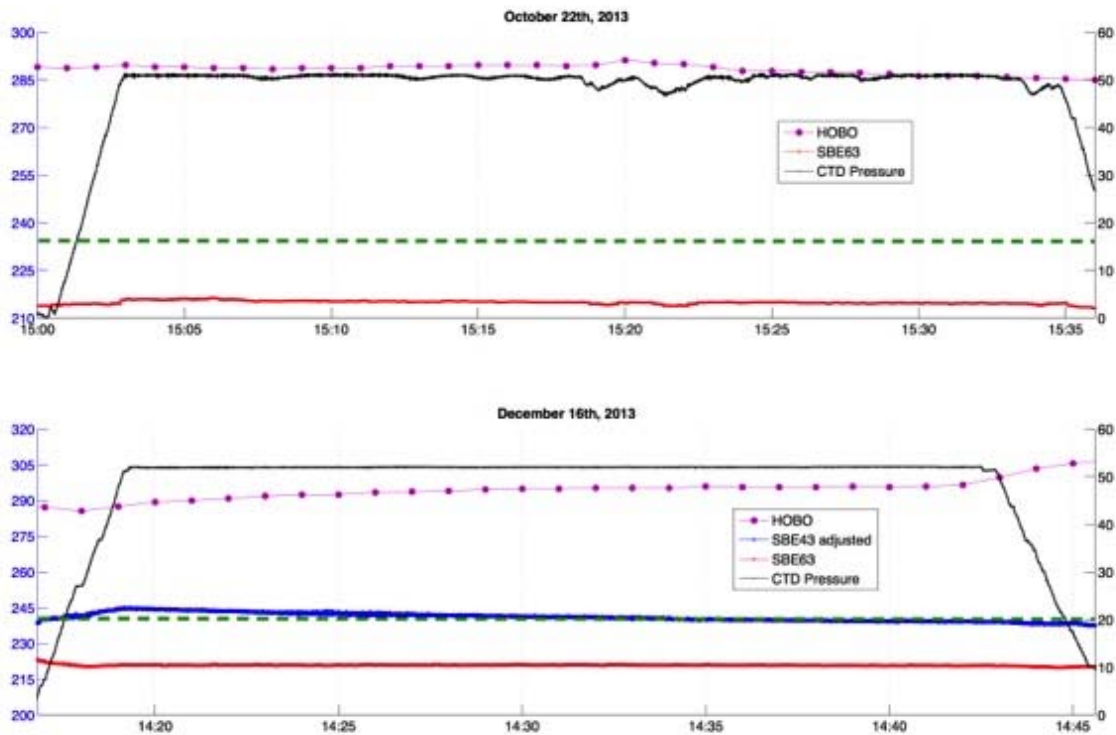


Figure 7: Oxygen measurements from HOBO compared to SBE43 and SBE63 sensors deployed in October 22th and December 16th. The dashed green line represents the Winkler reference measurements (234 and 241 $\mu\text{mol/kg}$, respectively)

The HOBO sensor from ONSET (optical sensor) was compared to the two SBE sensors and the *in situ* Winkler measurements. It's obvious that the HOBO shows a large offset of 70 $\mu\text{mol/kg}$ compared to the oxygen reference value (Winkler) due to the low accuracy of the sensor (0.2 mg/L = 6 $\mu\text{mol/kg}$), the low cost factory calibration and probably the too long lasting calibration procedure. However, even the frequency acquisition of the HOBO sensor is less than the SBE sensors (one minute instead one second for the SBE sensors), the stability of the O₂ signal seems to be adequate for long-term measurements in coastal areas where O₂ concentrations are not limited.

3.3 Conclusion and recommendation

The use of oxygen sensors in coastal marine environments are clearly more depending on biofouling issues than detection limits (more sensitive in open ocean areas and oxygen minimum zones). The field experiment performed here aimed to test the reliability and the accuracy of oxygen sensors response versus the *in situ* oxygen measurements (Winkler method). The results showed that: 1) regular calibration should be operated on each way or in order to provide accurate values, 2) optical sensors are less sensitive to biofouling and cleaning issues but require at least a 2 years calibration, 3) the correction applied to the new SBE63 sensor needs to be more documented



by the manufacturer. In this case, we can established a list of performance for each sensors according to their capacity (+++ good, ++ medium, + bad):

	SBE 43	SBE 63	Aanderaa 3975	Aanderaa 4330	HOBO U26
Accuracy	+++	++	+	++	+
Long term reliability	+	Not tested	+++	+++	++
Time response	+++	++	++	++	+
Data correction method	+++	+	++	++	+
Easy to use	+	+	+	+	+++
Costs	+	+	++	++	+++



4. Second FCT workshop

Title:

Dissolved Oxygen calibration / What are the best procedures?'

An interactive workshop to identify the best practices about dissolved oxygen calibration proceed

4.1 Workshop goals

Following the recommendations of the first workshop about organising short workshops focused on specific topics, application or technology, the second FCT workshop was focused on DO sensor calibration. Indeed, this issue remains a critical stage to produce quality and meaningful data for oceanographic studies. The second FCT workshop intended then to gather scientists, manufacturers and users to share and discuss their sensor calibration procedures with the idea to initiate the writing of a guide on best practices to calibrate oxygen sensors that would be a valuable document for both users and manufacturers.

4.2 Organization of the workshop

The second FCT workshop was organized at Oceanology International 2014 (OI2014) as this event brings together most of the oceanographic institutes, sensor manufacturers and users. This one-day workshop was divided in two sessions according to the following agenda.

In the first session (morning), participants shared their knowledge with presentations of their calibration protocols and how they carry them out. Laboratories for standard and reference measurements shared also their feedback and expertise in the domain of certification and Environmental Technology Verification (ETV) schemes. Several companies and sensor users (from surveillance to monitoring) presented their expertise/feedback as well as their needs for their day-to-day field activities.

The second session (afternoon) consisted of a moderated discussion about questions and topics raised during the morning session i.e. calibration, maintenance, long term stability, calibration market, organization, cost and know how. Information and data to harmonize and improve the calibration procedures were discussed.



About 30 people attended the workshop. Almost half of the attendees (40 %) originate from private companies; mainly sensor manufacturers and DO sensors suppliers were present. The workshop also attracted representatives from the main European oceanographic institutes carrying out research in coastal waters.

4.3 Presentations

The presentations given during the workshop are available on Jerico's website (www.jerico-fp7.eu/coastal-technologies/fct-workshop-2).

4.3.1 General introduction

As Jerico FCT manager, Glenn Nolan from the Marine Institute (Ireland), opened the session with an overview of the terms of reference and the main objectives of the FCT. He highlighted the meaning and the benefit of industry involvement in European projects like Jerico. He also presented the Jerico scheme and its interaction with other European oceanographic projects. This presentation showed clearly that opportunities exist for companies to promote their development and new technologies. However, necessary and regular exchanges of information are required about both technological developments carried out by companies and user requirements.

4.3.2 Calibration needs and current practices

Florence Salvetat (head of calibration laboratory, Ifremer) set out the calibration needs, the current practices and the potential alternatives. A clear distinction has to be made between coastal waters and open ocean; the accuracy requirements are substantially different ranging from 1 $\mu\text{mol/l}$ to 6 $\mu\text{mol/l}$ of DO. The user needs must then drive any calibration operations. The current practice is to carry out a 'multi-points calibration' to control the sensor linearity using Winkler titration.

A description of the bubbling systems was done, with specific bench designed for optical sensors (optode). Whatever the quality of the calibration process is, the accuracy is still about few $\mu\text{mol/l}$.

This presentation raised different topics for discussion:

What are the user needs?

How much are people ready to pay for appropriate calibration?

How to make in situ calibration?

How to use low cost sensor (statistical processing)?

How to be more efficient (training, transfer of know-how)?

Continuous and portable measuring devices, standardisation, certification or ETV verification?



Nathalie Guigues from the French National Metrology and Testing Laboratory (LNE) described the 2 main water quality standards ISO TC147 and CEN TC. The laboratory testing (in controlled environment) must be completed with field testing to be sure that claimed performance is not degraded in real application. The standard requires a huge set of performance verification.

In the road of acceptance of new sensor or measurement protocols, there are 3 main steps: standardization, certification and ETV verification.

The presentation described certification processes that should promote public confidence in data. For the sensor developers, it is a framework to guarantee that measurements are carried out in accordance with regulation's quality requirements. It is thereafter the role of environmental agencies to propose schemes to test and certify equipment for specific monitoring (eg MCERTS certification of DO sensors). ETV (European environmental Technology Verification) has been cited as a pilot program launched at European level. Any claimed performance can be verified.

4.3.3. COSYNA: Experiences with DO sensors

Wilhelm Petersen from the Helmholtz-Zentrum Geesthacht (Institute of Coastal Research, Germany) presented COSYNA, a coastal observing system for Northern and Arctic seas (fixed and mobile platforms)

Different types of DO sensors are currently tested in this infrastructure i.e. fast galvanic micro sensors and optical sensors. The protocol of long-term operation was widely described. Helmholtz-Zentrum Geesthacht uses an in-house calibration unit that is able to make calibration at different temperature and oxygen levels. Fast optical sensors are a good alternative to galvanic sensors while optodes reveal quite stable calibration in long term operations like ferry box. The problem of recalibration is not fully solved.

4.3.4 Oxygen measurement: sensors accuracy and scientific needs

Laurent Coppola from Observatoire Océanologique de Villefranche/Mer (CNRS – INSU, France) gave, on behalf a group of scientists, an overview of the scientific needs.

DO measurements present many interests for the understanding of the behavior of our oceans as DO measurement is one of the most important parameters giving information on the environmental and biological conditions of the oceans (eg. ventilation, convection, water mass age, organic matter remineralization and bacteria respiration). DO measurements help to understand water circulation, but also give an estimate of primary production (net community production) and is also a sensitive tracer for global climate change.

A summary of the sensors available on the market, with their real characteristics was presented. It appears that two type of sensors, electrolyte and optical technology, are mostly used. The performances in the field environment are compared.



It was shown that all stages of sensor life influence the quality of measurements (calibration, storage, test before deployment, quality check after deployment).

4.3.5 Improved oxygen optode measurements

Anders Tengberg from the Norwegian company Aanderaa Xylem presented advice on how to improve oxygen optode measurements. User examples, practical handling and calibrations were presented. Oxygen optodes have many advantages: long term stability, resistant to bio fouling, no pressure hysteresis, not sensitive to H₂S, low noise. Used on Argo floats, optodes showed stable results even if they gave slightly lower values with regard to standard probes. Main reasons evoked were foils bleaching in ambient light (use black cap when storing).

About calibration, using an in house multipoint calibration system, the supplier guarantees accuracy better than 1.5% +/- 2.5 µmol/l.

Finally, plastic materials that have "memory" effects on O₂ should be avoided and sacrificial anodes can induce artifacts as they consume oxygen.

4.3.6 Fast response DO sensor(JFE)

Hua Li, from JFE Adventech Co Ltd (Japan), talked about the response time of DO optical sensors. Rinko is a range of autonomously fast response optical DO sensor that, compared to other optical sensors, give a very fast measurement (90% response time is about 1 second). This allows fast and continuous profiles.

Calibration is done in house in two point 0 and 100% oxygen saturation after a warming up delay.

4.3.7 Monitoring Dissolved Oxygen: calibration and validation (Sea Bird)

Norge Larson, from Sea-Bird Scientific (USA), gave a presentation about monitoring Dissolved Oxygen: calibration and validation. In particular, he showcased the electrochemical sensor SBE 43 and the optical oxygen sensor SBE63 with their new features such as fast response time, reduced drift, not sensitive to H₂S poisoning. These sensors are respectively designed for fast and slow profiling uses.

The two DO sensors are plumbed (protected from external environment) and pumped along with CTD sensors.

Sensors are calibrated at multiple oxygen concentration, temperature and pressure points. Sea-Bird advises to follow the manufacturer's guide on maintenance and storage. Pre deployment checks and comparison between sensors are recommended. The company offers complete calibration and service in Europe.



4.3.8 Monitoring Dissolved Oxygen: calibration and validation (NKE)

Damien Malarde represented NKE Instrumentation (France), a company that designs, manufactures and sells instruments for the measurement and the monitoring of oceans. He gave a short presentation about calibration and validation. NKE usually makes in house 2 points calibration.

4.4 Moderated discussion

The discussion started with some words on metrology and semantic issues e.g. uncertainty is different to repeatability. It was mentioned that the uncertainty on Winkler titration (which is the unique reference) due to salinity is $< 4 \mu\text{mol/l}$.

A better collaboration between the laboratories that perform calibration is needed. It was suggested that an application to the European call COST could be initiated. The attendees agree that discussion and exchange between calibration laboratories should be enhanced.

Uses and calibration procedures were discussed by companies. The latter gave their recommendations for their instruments to reach the accuracy needed by scientists. Usually multipoint calibrations are carried out by the supplier before sensors enter the market. Then the end users should regularly check this calibration and not forget correction of temperature effect that is particularly important. A point of calibration must be done (instrument in service) when coming to the surface (profiler float), one point in the atmosphere with a very accurate measure of temperature in gas phase. This action allows a drift correction.

Companies offer the service of their calibration laboratories (open in Europe close to their customers). All the sensors are returned to the same place, where the laboratory can guarantee that the operation behaves properly. The laboratory delivers the whole sum of uncertainty. After cleaning, following operation, sensors are calibrated again with the same protocol.

The service protocol is applied to the whole life of the sensor including the storage before deployment (follow manufacturer' guidance). For optode's sensor, foils bleach when exposed to ambient light.

Manufacturers also expressed their needs to obtain a consensus on practice (or standard) to standardize the protocols. Beginning within Jerico, few and now more users carry out calibration linked with manufacturers. These companies could participate to the standardization process (at ISO level that means users will manage the whole process). Jerico could help to achieve and succeed in this challenge. In particular, it should help to build capacities, gather information and data to share a best practices guide.



A recommendation was made that a reference laboratory could organize and manage a working group but means (funding and human resources) are needed. Manufacturers agree to join such an initiative. Existing work funded by NOAA in 2012 about the quality control of DO measurements could be used as a starting point. Thereafter the writing of a guide will help to sustain the network as well as questions raised could be shared and answered on a dedicated website (perhaps within next Jerico project). At this stage of the discussion, it was mentioned the French initiative of the SOMLIT network (CNRS) that conducts yearly instrument comparisons, in the same waters following a shared protocol.

Issues about training and audit are raised by LNE. Collaboration with oceanographic institutions should be initiated to help to set up and organize a training program. In the meantime, some companies such as SeaBird, have built their own calibration capacity, in strong collaboration with universities and scientists. They offer training to DO sensor users. They say that this solution is better than national standard, but the expertise can be specific to their own sensors. They are interested in inter-laboratory comparison to validate calibration protocols.

Finally, the question of low cost sensors was also treated even if it appears not well accepted by the scientific community. The main reason for that is the confidence in the generated data is very low. At this stage, the market for this kind of sensor, based on new technology and new deployment protocol must be assessed. Applications might exist in industry such as aquaculture or water quality control to address new regulation.

The afternoon session was finally summarized and concluded by **Patrick Farcy**, (Jerico project coordinator) highlighting that Jerico will support the COST action proposal, to build a European calibration capacity. He also mentioned that issues on data quality and uncertainty will be central to other (and future) European projects. New projects such as ATLANTOS, FIXO₃, NEXOS are focused on the development of new sensors (or new technology for sensors), the comparison between measurement protocol and methodologies (and calibration as well). Finally, the question of the funding of a EUROACT or equivalent for coastal technologies is still pending.





Annex 1: Workshop agenda

‘Dissolved Oxygen calibration / What are the best procedures?’

An interactive workshop to identify the best practices about dissolved oxygen calibration procedure

2nd Forum for coastal technologies Workshop AGENDA

	Item	Speaker
9 30	Welcome and Introduction	Glenn Nolan Marine Institute (Ireland)
9 50– 12 30	Session One - Presentations	
9 50	Scientist focus Calibration needs, current practices and alternatives	Florence Salvetat, IFREMER (France)
10 10	Certification organism focus Performances of continuous and portable measuring devices: standardization, certification or ETV verification?	Nathalie Guigues, Lab. National de Métrologie et d'Essais (France)
10 30	Scientist focus Feedback on oxygen sensor calibrations (AADI's oxygen optodes, JFE's RINKO) and long-term stability in FerryBox systems	W Petersen HZG - Helmholtz-Zentrum Geesthacht (Germany)
10 50	Scientist focus Oxygen measurements in oceanography: sensors accuracy and scientific needs	Laurent Coppola Obs. de Villefranche-sur-Mer / CNRS-UPMC (France)
11 10	Manufacturer focus Improve your oxygen optode measurements - user examples, practical handling and calibrations	Anders Tengberg, Aanderaa (Norway)



11.30	Manufacturer focus Fast response DO sensor - RINKO	Hua Li JFE Advantech Co., Ltd (Japan)
11 50	Manufacturer focus Monitoring Dissolved Oxygen: Best Practices, Calibration, and Validation	Norge Larson Sea-Bird Electronics (USA)
12 10	Manufacturer focus Views and experience on the implementation of dissolved oxygen calibration at NKE instrumentation	Damien Malardé NKE Electronics (France)
	Break (lunch)	
14 – 16	Session Two	
14 00	Facilitated Discussion Topic #1: Adapted calibration (coastal or open sea) and the essential calibration steps (good practices) Topic #2: Calibration Market (low cost sensors, training, certification, QC...) Topic #3: Main 'field' vs lab issues Restitution and synthesis	Chaired by: Yannick Aoustin IFREMER (France) (Audience might be divided in three groups)
1600	Summary & close	Patrick Farcy (Coordinator) IFREMER (France)



Annex 2: List of attendees

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