Joint European Research Infrastructure network for Coastal Observatory – Novel European eXpertise for coastal observaTories - **JERICO-NEXT**	
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Coordinator	P. Farcy	Ifremer	11/09/2019	PF
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1. Executive Summary

This document provides an overview of task 2.5 (“Calibration and assessment”) of Work Package 2 (“Harmonization of technologies and methodologies - technical strategy“) of JERICO-NEXT, summarizing its different activities during the project’s lifetime.

The task aimed to lay down the foundations for a coordinated calibration and assessment system for the JERICO observing network in JERICO-NEXT. It gathered together 12 partners from 11 European countries (France, Italy, Germany, Greece, Norway, UK, Portugal, Sweden, Spain, Sweden, and Finland), and was co-led by two of them, Ifremer and OGS.

2. Introduction

The reliability of an observing network is dictated by its ability to consistently deliver usable information, meaning that the data generated by the network must be comparable, traceable, and of ascertainable quality at all times. Acknowledging that methodical calibration and assessment activities, supported by relevant metrological expertise wherever possible, are basic steps to meeting these requirements for gathered data, JERICO-NEXT envisioned a specific task, task 2.5, which was aimed at laying down the foundations for a coordinated calibration and assessment system for its observing component, the JERICO coastal observatory network. The task, entitled simply as "Calibration and Assessment", was conceived as part of Work Package (WP) 2 ("Harmonization of technologies and methodologies - technical strategy"), the WP dealing with network harmonization, where it would be contributing to the following:

- the standardization of measuring practices;
- the reconciliation of technological and operational differences relating to measuring activity.

In the light of the budget that was finally allocated to the task, these objectives were of necessity condensed into a very limited series of activities:

- the organisation of two workshops to support the activities of the task during the project;
- a small performance evaluation (PE) exercise for salinity measurements across the network;
- two small inter-laboratory comparisons, one for the measurement of dissolved oxygen and another for the determination of pH in seawater matrices;
- the investigation and documentation of the general challenges and developmental needs for the calibration of optical sensors for biology-related measurements, with information coming from some laboratory testing aimed at harmonising methods for calibrating fluorometers;
- the formation of a Calibration Board to oversee matters relating to calibration and metrology within the JERICO RI, based on the one constituted during the preceding FP7 JERICO project.

3. Main report

3.1. The task 2.5 "Calibration and Assessment" workshops

The first workshop of Task 2.5 on calibration and assessment was held in Brest, France, on the 11th and 12th of October, 2016. The workshop served to present, describe and discuss the actions planned within the task, including their links with the other tasks and work packages (especially those relating to sensors and systems) of JERICO-NEXT. The main topics addressed were the metrological consistency of observations, and the comparability and quality of data and data products. The event provided the first occasion for the participants in the task to meet and interact with each other directly since the start of the project, and review the outcomes of similar actions that were undertaken during the preceding FP7 JERICO project, which had ended in 2015, to try and draw useful lessons and parallels.

The agenda of the workshop is included below (table 1) to outline the main topics that were addressed.

Tuesday, 11 October 2016		
Time slot	**Title**	**Speaker**
13:30 -13:45	Introduction of JERICO-NEXT. Need of metrology, its impacts in the project.	P. Farcy
13:45 -14:15	Metrology TNA Facilities in JERICO-NEXT.	F. Salvetat/M. Ntoumas/ J.Seppala
14:15 -14:45	What is metrology: from the sensor calibration point of view to the need of a proper definition of the measurand and the help in exploiting and valorizing data.	F. Salvetat
14:45 -15:00	Links with the other tasks.	R. Nair
15:00 – 15:45	2 to 3 examples from the scientific community relevant to metrology problems or need of harmonization.	M. Ntoumas/J. Seppala
16:15-16:45	Previous metrology actions in JERICO-FP7.	M. Ntoumas/G. Petihakis
16:45 – 18:00	Flash presentations / Discussions / Questions (harmonization, sensor performance in the different environmental conditions between regions → support from metrology).	Various
End of first day		

Wednesday, 12th October 2016		
Time slot	**Title**	**Speaker**
9:00-9:30	JERICO-NEXT actions planned – the Salinity PE Exercise: What was done so far and next steps (participants, devices, organization).	F. Salvetat/R. Nair
9:30-10:00	JERICO-NEXT actions planned - pH ILC: pH issues.	D. Stoica
10:00-10:30	JERICO-NEXT actions planned - pH ILC: pH exercise.	F. Salvetat/R. Nair
11:00-11:30	JERICO-NEXT actions planned - JERICO-NEXT Label (Calibration "network"/roadmap/strategy).	G. Petihakis
11:30-12:30	Summary and closing.	F. Salvetat/R. Nair
14:00-17:30	Visit of Ifremer facilities (40 pers.)	
End of Workshop		

Table 1. Agenda of the first workshop of Task 2.5 (WP2) on calibration and assessment.

The main outcomes of the workshop were the following:

- a mapping of the effective metrological resources available within the JERICO community;
- strong consensus on the necessity to keep on working towards the sharing of experiences and harmonization of practices for salinity, pH, O_2 and fluorescence measurements;
- broad agreement on the necessity to have more partners involved in the issues relating to calibration and assessment;
- only 8 partners out of the 17 represented seemed to be concerned about metrology, highlighting the difficulty in showing the importance of addressing/valorising metrology within the oceanographic research community;
- progress on the actions involving inter-laboratory comparisons planned in the task (the declaration of willingness by partners to participate, discussion of technical protocols, etc.), coloured by the general interest evidenced to focus on activity in the field.

The second workshop of Task 2.5 on calibration and assessment was held in Brest, France, on the 11th of October, 2018, as part of the joint EMSO ERIC – JERICO-RI - AtlantOS workshop on "Interoperability Technologies and Best Practices in Environmental Monitoring" (Brest, France, 10-12 October 2108). The workshop served to present, describe and discuss standing metrology-related issues concerning dissolved oxygen, pCO_2 and pH measurements in the marine environment. The following topics of discussion were discussed:

- interoperability and metrology (what do they mean?);
- metrology tools, laboratory or/and on board equipment and protocols;
- metrology for carbon fluxes and acidification (are absolute in situ measurements achievable?);
- metrology for carbon fluxes and acidification (is traceability a dream?);
- traceability management;
- possibilities and performance of the EMSO O_2 calibration bench, and associated best practices that could be proposed;
- R&D needed for sensors.

The main outcomes of the workshop were the following:

- acceptance by the participants of the close link between the interoperability of technologies and metrology because of the centrality of the calibration process for all measuring devices;
- strong consensus on the necessity of a community approach to address the above link;
- broad agreement on the necessity to support standardization of measurement activity (procedures, Best Practice, reference material, etc.);
- acknowledgement of the need to develop actions to promote "marine" metrology at national and international levels;
- acknowledgement of the need for more work on the validity of in-situ measurements;
- overview and future prospects of the EMSO O_2 calibration bench as a metrological tool for dissolved oxygen measurements.

3.2. The Salinity Performance Evaluation (PE) Exercise

The JERICO-NEXT salinity performance evaluation exercise was jointly organized by the Task 2.5 leaders, Ifremer (France) and OGS (Italy) in 2016, as a first attempt to evaluate the comparability of salinity measurements across the JERICO observing network.

First, a registration form was prepared, and the laboratories making salinity measurements in the network were invited to fill and submit it as a formal indication of their intention to take part in the exercise. Once the registration process was completed, the number of participating laboratories was established and arrangements were made to deliver Performance Evaluation (PE) samples for the analysis of salinity to

them. All in all, 12 laboratories from 11 institutes in 10 countries (figure 1) participated. Nearly all of the laboratories were using bench salinometers standardized using IAPSO seawater standards for their primary salinity measurements. 10 of the salinometers involved in the exercise were products of Guildine instruments (7 “Autosal” and 3 “Portasal” units), and one was manufactured by Optimare.

The PE samples were prepared and sent out by Ocean Scientific International Ltd. (OSIL), the company based in the UK which produces the IAPSO seawater salinity standards. Each participant received three 200 ml glass bottles containing samples with different nominal salinities: circa 20, 30 and 38 (PSS-78). The participants were asked to determine the unknown salinities of these samples, preferably utilizing their main laboratory reference instrument for the measurand. It was highly recommended that the PE samples be included with other salinity samples in a routine analytical run.

The final deadline for the submission of measurement results was set for 01 March 2017. The results were to be reported to OSIL as well as to Ifremer and OGS.

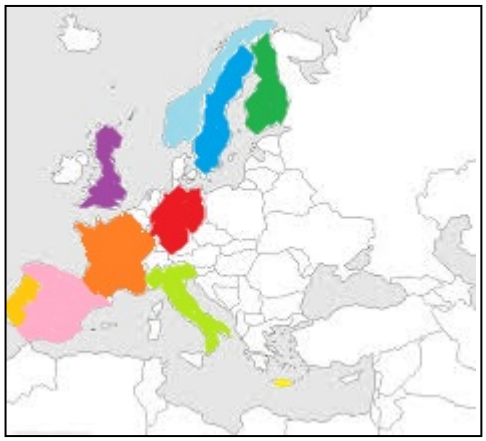
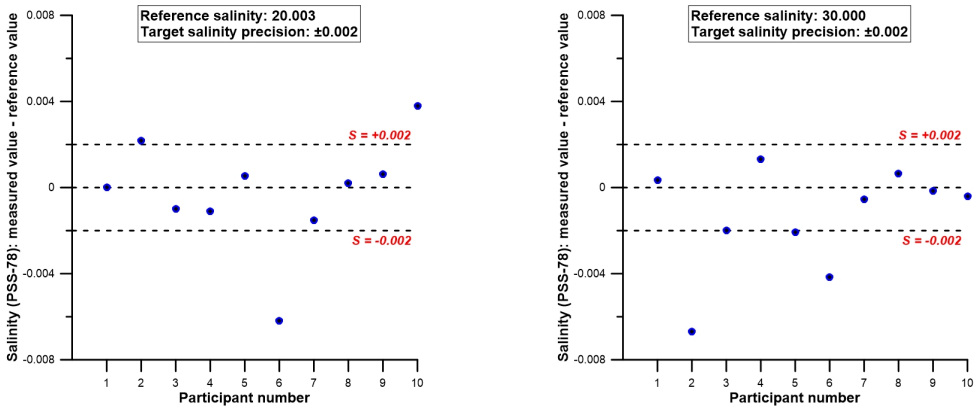
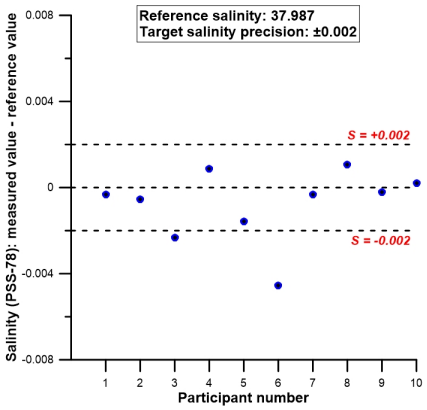


Figure 1. Map showing the countries represented in the JERICO-NEXT salinity Performance Evaluation (PE) exercise.

The results of the PE exercise are shown in Figure 2 below. Only 10 of the 12 participating laboratories are represented. One laboratory was not able to perform the measurements, and the results submitted by another have not been included because of some doubt regarding their validity owing to the way in which the samples were analysed.



(a) (b)



(c)

Figure 2. Results of the Task 2.4 salinity Performance Evaluation (PE) exercise: analysis results at nominal reference salinities of 20 (a), 30 (b) and 38 (c).

The control limits for the exercise were set to ±0.002 (PSS-78) with respect to the certified reference values of samples. This is equivalent to the manufacturer-declared accuracy for the Guildline 8400B bench salinometer, a recognized community standard for the salinity measurement. For the nominal reference salinity of 20, 7 out of the 10 laboratories considered submitted values that fell within these limits. A similar result was obtained for the samples with the nominal salinity of 30, also. Finally, 8 out of the 10 laboratories submitted satisfactory responses for the analyses corresponding to the nominal reference salinity of 38. Note that in all three cases, the "erring" laboratories were not always the same ones.

3.3. The inter-laboratory comparison exercises

3.3.1. Dissolved oxygen

The organisation of the inter-laboratory comparison exercise for dissolved oxygen (O_2) measurements was first discussed in 2016 during the workshop of Task 2.5 on calibration and assessment that was held that year. Two possible approaches were proposed for conducting the exercise: a comparison of either the performances of O_2 sensors belonging to participants or the abilities of participants in performing the "Winkler" method that is conventionally used for obtaining reference dissolved oxygen measurements. The Ifremer - Centre de Brest was chosen as the location for carrying out the exercise. It was decided to carry out a survey during 2017-2018 to understand which of the two possibilities was more attractive to the partners in order to make the final choice.

Ultimately, attempts towards the end of 2018 to finally carry out the exercise were hindered by difficulties in obtaining convenient dates for the necessary access to the Ifremer facilities and the poor adhesion of partners. The activity was therefore abandoned, in part also due to the impending close of the project in August 2019.

3.3.2. pH_T

The pH_T (pH on the "total" hydrogen ion concentration scale) inter-laboratory comparison exercise was proposed as a continuation of, and as a complement to, the other activities relating to the measurement of seawater pH in the project (the TNA "INTERCARBO", the pH roadmap of the JPI OCEANS EMARCALNET action). Interested partners were sent TRIS buffer solutions at a specified salinity (S=35) in order to evaluate the comparability of pH_T measurements. Two buffers were prepared, one with a pH_T of around 7.6 and another with a pH_T of around 8.2.

The innovation proposed in this interlaboratory comparison was to provide Certified Reference Material (CRM) in the form of buffers with traceability to primary standards. Each buffer was qualified and delivered with both a measured pH_T reference value and its associated uncertainty. The buffers were prepared and certified by the LNE, the French national metrological institute.

Five laboratories registered for the exercise, which was held in May 2019. Three of these have partially submitted their results (table 2), but the remaining two still have to do so.

	Standard value	+/- expanded uncertainty (k = 2)	Standard value	+/- expanded uncertainty (k = 2)
	7.625	+/- 0.005	8.211	+/- 0.005
	Measured value	+/- standard deviation	Measured value	+/- standard deviation
Lab 1	7.638	0.002	8.221	0.002
Lab Error	0.013		0.010	
Lab 2	7.629	0.001	8.201	0.002
Lab Error	0.004		-0.010	
Lab 3	7.629	0.003	not reported yet	
Lab Error	0.004			
Lab 4	not reported yet		not reported yet	
Lab Error				
Lab 5	not reported yet		not reported yet	
Lab Error				

	Standard value LNE	Standard value Dickson	Standard value LNE
	7.625	7.836	8.211
Lab 1	7.638	7.838	8.221
Lab Error	0.013	0.001	0.010

	Standard value LNE	Standard value Dickson	Standard value Dickson	Standard value LNE
	7.625	7.833	8.093	8.211
Lab 2	7.629	7.815	8.092	8.201
Lab Error	0.004	-0.018	-0.001	-0.010

	Standard value LNE	Standard value Dickson	Standard value LNE
	7.625	8.043	8.211
Lab 3	7.629	8.043	-
Lab Error	0.004	0.001	-

Table 2. The Task 2.5 pH_T inter-laboratory comparison exercise: the pH_T values assigned by the LNE to the Certified Reference Material (CRM), and the submitted measurements of the same by participating laboratories along with the respective errors.

The first results show that trueness errors can reach as high as 0.013 of pH_T which is greater that what is usually expected (<0.01). These errors can be compared to the values of the adjustments that are made when controlling the trueness of pH_T measurements against the Dickson pH_T standards commonly used for this purpose.

The results also clearly indicated a difference in trueness depending on the use of one or the other CRM figure 3). Why this was so has still to be investigated.

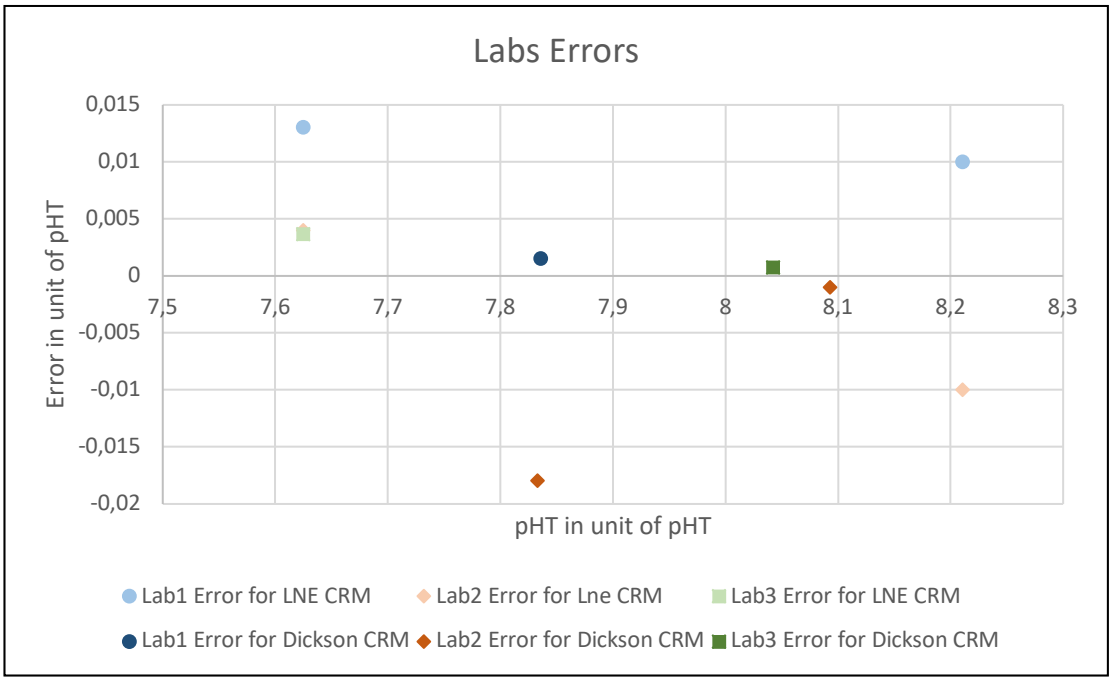


Figure 3. The Task 2.5 pH_T inter-laboratory comparison exercise: laboratory errors of measurements for the LNE and Dickson Certified Reference Material (CRM).

3.4. Fluorometers: calibration and comparability of measurements

The calibration of fluorometers for measuring chlorophyll-*a* fluorescence has been the subject of major discussion in several JERICO-NEXT workshops, not only in those relating to task 2.5 and the other tasks of WP2. The state-of-the-art as regards calibration for various kinds of optical sensors using a variety of techniques (LED fluorometry, spectral fluorometry, fluorescence induction, spectral absorption, spectral reflectance, turbidity and scattering, and pulse shape-recording automated flowcytometry) is described in the JERICO-NEXT deliverables D2.2 ("Report on new network sensors") and D2.5 ("Report on Best Practice for new network sensors), which can be accessed through the project website (www.jerico-ri.eu/project-information/deliverables/).

Furthermore, in the case of fluorescence-based chlorophyll-*a* measurements, a dedicated workshop was organised to discuss this topic in detail (the joint JERICO-NEXT / AtlantOS workshop entitled "Interoperability of Technologies and Best Practices: in situ applications to nutrient and phytoplankton fluorescence measurements", Brest, France, 04-06 December 2018), and a white paper based on the resulting deliberations is under preparation. In addition, the standard operating procedures developed at the Finnish Environment Institute SYKE for calibrating fluorometers for making such measurements have been divulged to assist in harmonizing the practices employed for this operation within the JERICO-NEXT community.

The current main challenges encountered in dealing with fluorescence-based chlorophyll-*a* measurements have been identified as the following:

a) there is no commonly-agreed methodology for traceable primary calibration of field fluorometers for chlorophyll-*a*;
b) chlorophyll-*a* measurements made with fluorometers produced by different manufacturers are not directly comparable due to dissimilarities in the optical setups;
c) while the conversion factor between fluorescence and chlorophyll-*a* concentration, which can vary due to the physiology and community structure of phytoplankton and environmental conditions (e.g. light and nutrient availability), is typically determined case-by-case using field samples collected specifically for this purpose, the exact procedures employed are seldom reported;
d) there are no widely-shared Best Practices relating to Quality Assurance (QA) for chlorophyll-*a* data obtained with field fluorometers, especially from the standpoints of the primary calibration, the optical setup, and the fluorescence-to-chlorophyll-*a* conversion step;
e) as a consequence of all of the above (points a, b, c and d), fluorescence-based chlorophyll-a data stored in different databases may often not be consistent and comparable.

3.5. The Calibration Board

The Calibration Board of JERICO-NEXT was the same one constituted during the preceding FP7 JERICO project so as to maintain continuity and carry forward relevant initiatives relating to calibration and metrology begun then. The composition of the Board is shown in table 3, below.

Table 3. The JERICO-NEXT Calibration Board.

Name and surname	Affiliation
Laurent Coppola	*CNRS*
Rajesh Nair	*OGS*
Manolis Ntoumas	*HCMR*
Wilhelm Petersen	*HZG*
George Petihakis	*HCMR*
Ingrid Puillat	*IFREMER*
Florence Salvetat	*IFREMER*
Laurent Delauney	*IFREMER*
Jukka Seppala	*SYKE*

The Board was actively involved in all the activities of task 2.5, and indirectly in some activities of WP3 (areas: imagery instrumentation, HF-radar, profiling systems, combined sensors for the marine carbonate system) and WP5 (areas: HF-radar data, data relating to biology based on optical measurements, data on marine carbonate system variables, data from gliders, and platform registration and metadata management). It has also collaborated, either together or through its single members, both at the national level and in many national and international projects and programmes (e.g. JPI Oceans, AtlantOS, EMSO, the UNESCO-IOC Ocean Best Practices System, etc.).

4. Outreach, dissemination and communication activities

JERICO-NEXT, First workshop of Task 2.5 on calibration and assessment, Brest, France, 11 - 12 October, 2016; targeting mainly the JERICO-NEXT community but left open for eventual participation by other interested parties.

JERICO-NEXT, Second workshop of Task 2.5 on calibration and assessment, Brest, France, 11 October, 2018, held as part of the joint EMSO ERIC - JERICO RI - AtlantOS workshop on "Interoperability Technologies and Best Practices in Environmental Monitoring" (Brest, France, 10-12 October 2108); targeting mainly the JERICO-NEXT, EMSO ERIC and AtlantOS communities.

White paper, tentatively entitled "Interoperability of Technologies and Best Practices: in situ applications to phytoplankton fluorescence measurements"; publication in preparation for submission, (preferably) to an open-access journal to permit the widest possible dissemination.

5. Conclusions

The result of the salinity PE exercise evidences the need for constant vigilance to ensure the comparability and quality of observed data, especially in a networked environment. The measurement of salinity with a bench salinometer is a well-established procedure, widely assumed to be reliable and free from problems. The dispersion noted in the values of the differences is therefore enlightening, especially if one considers the relatively small number of respondents and the uniformity in terms of the measuring instrument used for analysing the samples. The question that merits reflection in the light of the result of the exercise is that, if this is the situation for salinity, then what about all the other observables that are being measured?

In the case of dissolved oxygen (O_2) measurement, while there is general acceptance that the reference method is the Winkler method, a number of difficulties remain:

- the Winkler method is operator-dependent, and requires great experience in carrying out both the sampling and the analysis in order to be reliable.
- reference material - i.e. O_2 standards - are lacking (leading to the necessity to organize and to participate in inter-laboratory comparisons, preferably at sea - not an easy task at the best of times).
- O_2 measurements made with the Winkler method often come without any uncertainty information.

The topic of quality pH measurements is currently a big concern in the oceanographic community. Several basic issues need to be tackled in this regard. There exist several pH scales not all referring to the same measurand and, therefore, the traceability of measurements is a matter for debate. Furthermore, depending on the technology that is used to measure pH in the field (spectrophotometry or potentiometry, for instance) or on the calibration procedure adopted (type of standards employed: IUPAC or total pH), different quantities are estimated (the concentration of either free protons or free protons plus the protons associated with sulphate). The wrong choice of standard can lead to discrepancies on the order of several hundredths of pH (>0.01). There is, therefore, a pressing need to investigate the capacity of laboratories in performing traceable, spectrophotometric pH measurements, which are usually used as reference values, through sustained inter-laboratory comparisons with a strong metrological component.

Thus, it is clear that metrology should always be an integral part of the process of acquiring data, and must be considered right from the beginning of any observing activity or, in the case of technology, during the very first steps of the design of a sensor and the choice of its measuring technique. Given the specificities of the marine environment from the standpoint of observables, technologies and operating

conditions, this means specialized knowledge, expertise and training. All of these are currently in short supply within the marine observing community at all levels, local, national, European and global. Finally, it will be necessary to build strong links and collaborations with the system of national metrological institutes in order to ensure that collected data comply with normative requirements so that they can be exploited by users in other sectors, not just in science and research where this need is less felt at the present time.

6. References

JERICO (2014). *Report on the constitution of a permanent JERICO Working Group for Calibration Activities*. Milestone (MS15), Project JERICO (Towards a Joint European Research Infrastructure network for Coastal Observatories; EU-FP7).

JERICO-NEXT (2017). *Report on the status of sensors used for measuring nutrients, biology-related optical properties, variables of the marine carbonate system, and for coastal profiling, within the JERICO network and, more generally, in the European context*. Deliverable, Project JERICO-NEXT (Joint European Research Infrastructure network for Coastal Observatory-Novel European eXpertise for coastal observaTories); EU-H2020), JERICO-NEXT-WP2-D2.2-28062017-V1.2.

JERICO-NEXT (2017). *Best practices for quality control of sensor based biochemical data*. Deliverable, Project JERICO-NEXT (Joint European Research Infrastructure network for Coastal Observatory-Novel European eXpertise for coastal observaTories); EU-H2020), JERICO-NEXT-WP5-5.11-061117-1.3.

JERICO-NEXT (2018). *Guidelines for the delayed mode scientific correction of glider data*. Deliverable, Project JERICO-NEXT (Joint European Research Infrastructure network for Coastal Observatory-Novel European eXpertise for coastal observaTories); EU-H2020), JERICO-NEXT-WP5-D5.15-140818-V4.0.

Van Ganse Sophie, Martinez Enoc, Salvetat Florence, Le Gall Christian, Del Rio Fernandez Joaquin, Toma Daniel Mihai, Niclas Chirurgien Laure, Lefevre Dominique, Ntoumas Manolis, Petihakis George, Coppola Laurent, Favali Paolo, Dañobeitia Juanjo, Blandin Jerome (2019). *EMSO-Link D2.2 - First release of handbook of Best Practices with two main chapters: Metrology and Sensor Web Enablement*. Deliverable, Project EMSO-Link; European Multidisciplinary Seafloor and water column Observatory (EU-H2020). EMSO-Link-D2.2_V2.1.