Joint European Research Infrastructure network for Coastal Observatories

հոհոհոհ



Report on existing calibration facilities D4.1

Grant Agreement n° 262584 Project Acronym: JERICO

<u>Project Title</u>: Towards a Joint European Research Infrastructure network for Coastal Observatories

Coordination: P. Farcy, IFREMER,

jerico@ifremer.fr, www.jerico-fp7.eu:

<u>Authors</u>: Michael Haller, Wilhelm Petersen, George Petihakis, Manolis Ntoumas, <u>Involved Institutions</u>: HZG, HCMR



ահանությունությո

Deliverable 4.1- date:22/01/2013

Table of Contents

TABLE OF CONTENTS	3
1. DOCUMENT DESCRIPTION	5
2. EXECUTIVE SUMMARY	7
3. INTRODUCTION	8
4. MAIN REPORT	10
4.1. General information	13
4.2. Calibration Facilities for Physical Sensors4.2.1. Temperature4.2.2. Salinity	26 26 29
4.3. Calibration facilities for Optical Sensors	30
4.4. Calibration facilities for Chemical Sensors	33
5. CONCLUSIONS	36
ANNEXES AND REFERENCES National Oceanography Centre Southampton	41 41

1. Document description

information at its sole risk and liability.

REFERENCES



Annex 1 to the Contract: Description of Work (DoW) version XX

Document	informa	ation						
Document	Name	me Report on existing calibration facilities						
Document ID		JERICO-WP4-D4.1-121226-V1						
Revision 1.								
Revision D	ate	2012-1	2-25					
Author		Michae	l Haller, Wi	lhelm Petersen, Geor	ge Petihakis, Manolis N	Itoumas,		
Security								
History								
Revision	Date		Modifica	ation		Author		
1.0	28/11	/12	First comp	blete draft version		mh		
1.1	07/12	/12	Modificatio	ons		wp		
1.2	13/12	/12	Additions			mh		
1.3	21/12	/12	Conclusions, additions			gp, mn, wp		
1.4	16/01/13		Revisions			mh		
1.5	22/01	/13	Minor revisions		wp, mh			
Diffusion li	ist							
Consortium beneficiaries			Х					
Third parties								
Associated Partners								
Neither this communica written cons	docume ted by a sent of th	ent nor any me he JER	the infor ans to a ICO Coo	mation contained ny third party, in rdinator.	brietary to the JER I herein shall be us whole or in parts, nd no guarantee or	ed, duplicated or except with prior		
	formatio	on is f	fit for an	iy particular pur	pose. The user the			



ատուսուս

Deliverable 4.1- date:22/01/2013

2. Executive Summary



The main goal of work package 4 of JERICO project is to increase the performance of JERICO observatories. The first step on this way is to evaluate existing calibration facilities of JERICO partner institutes.

So, this report is providing information about

- General features of calibration systems:
 - o Budget for calibration
 - o Calibration staff
 - Quality management, control charts, links and collaboration with other institutes
- Evaluation of sensor calibration specifications for
 - o physical sensors
 - o optical sensors
 - o chemical sensors

JERICO Calibration facilities are evaluated regarding their budget and staff as well as their overall reliability. An important issue for reliable calibration is the application of quality management standards, control charts, accreditation and links to other institutes. These applications are evaluated in this report as well.

The calibration routines are dependent on the applied sensor type, so we distinguish in this report between physical, optical and chemical sensors. We evaluate the overall reliability for different sensor type and we go also in detail about calibration intervals and details of the calibration settings.

At last, we draw conclusions about what is the latest standing of calibration systems among the JERICO partner institutes and what is supposed to be improved in the future and what has been suggested by JERICO partners.

lintintintintintint

3. Introduction

Operation and maintenance activities are probably the most crucial elements in the life-cycle of a research infrastructure and in some cases even more demanding than the design and construction of the infrastructure itself.

A sensor is only as good as its calibration, so a good sensor produces only poor results if the calibration is insufficient. Good sensors require both reliable sensors and reliable calibrations. The successful implementation of operation and maintenance activities guarantees the good performance of the infrastructure and the protection of the investment. Coastal observatories have been developed in Europe in a rather uncoordinated way. Usually based on national funding and priorities these observatories have very diverse design and architecture and have established very different practices for their operation and maintenance. For certain subsystems (e.g. FerryBox) past EU projects have established a network of operators through which experience and best practices have been shared but this is not the case for other observing platforms, and certainly not for integrated coastal observatories.

Therefore more work is needed to gather and combine information of relevant calibration issues.

Thus, one major task of Work package 4 (WP4) is to report on existing calibration systems which have been installed and are maintained amongst the JERICO partners. In terms of reliability it is evaluated which calibration features are applied by partner institutes. The main calibration features are financial budget, calibration staff, quality management and links to other institutes.



Information of best practise can be gathered, analysed and adopted to new calibration strategies to improve the performance and efficiency of oceanographic measurements of different sensor types in the future.

For the proposal of common practises it is necessary to combine work which is done in Work package 3 (WP3), as calibration matters are depending on the measuring platform. This is an issue evaluated by WP3.

lintintintintintint

4. Main Report

Reliable calibrations of instruments require well-established, documented procedures, specialized instrumentation, certified or recognized reference material (where these are available), dedicated laboratory facilities, trained personnel, and proven expertise. Although sensor calibration is absolutely crucial for good quality data, it is also a rather difficult task since different sensors have completely different requirements (e.g. time intervals) and methodologies. There are two major problems; shipping sensors to manufacturers on regular basis which is neither convenient nor cost efficient and maintenance intervals that have to be planned according to the requirements of each sensor (need for double sets of sensors). Thus transport and calibration costs often have a major contribution on total running costs. Although there is significant experience among European research institutes on calibration methods, at present each lab works independently with no or very little connections with other labs.

The JERICO activities will:

- standardize and harmonize various facilities across European networks,

- share existing calibration facilities within the network, thus significantly reducing costs

- exchange and transfer know-how within the network through a series of workshops, seminars and staff exchange.

l in hind in hind in deal



Figure 1 : Overview of JERICO partner institutes contributing to calibration questionnaire.

In this report we draw a picture of existing calibration facilities which have been established at several institutes being a partner in JERICO. Thus, a calibration questionnaire has been launched and distributed among the JERICO partners who run a calibration facility (see Figure 1). We got back 18 questionnaires. However, some of them are not completely filled in. So the total number of answers varies somewhat depending on the part of the questionnaire.

In Table 16 and Table 17 an overview of the questionnaire is presented. They show the questions and the corresponding numbering which will be referred to later on. For evaluating the answers to the different issues in the questionnaire, we use the term "reliability" as a measure for the quality of the calibration facilities. For part one of the questionnaire, institutions could get at maximum 16 points if they could answer in the positive each time which means best reliability.



.....

In part two regarding different sensors types, not all questions are usable for this kind of evaluation (see Table 17). Question (3) in the second part has been excluded from the evaluation of reliability as well as question (7) which is rather a question of opinion, if regular factory calibration and servicing is necessary to obtain optimal performances from your sensors for the specified parameter in the field. The question (2) and (5) requests for giving details of calibration routines (e.g. frequency of calibration, details of calibration setup) and, thus, is not usable for a reliability evaluation. Question (11) and (12) asks the institute's responsible person for ideas regarding the calibration quality of the used sensors.

Thus, the remaining questions (4,6,8,9.1,9.2,10) could be added up to define a reliability level of the calibration facility for the certain sensor type (marked in Table 17). A maximum of 6 reliability points can be achieved. This is then discussed later on for physical, optical and chemical sensors in the sections 4.2, 4.3 and 4.4, respectively.

C

Intration of the last

4.1. General information

The first part of the questionnaire deals with the general aspects of calibration issues such as facility staff for calibration (1), funding for the calibration facility (2) and other main features of the facility (3)-(12). Each question is listed in Table 16. Regarding staff issues, we wanted to know if staff is employed only for calibration (1.1), if there is a clear staff hierarchy (1.2) and if there is a transparent chain of responsibility (1.3). The highest level of reliability is reached by answering in the positive.

For question (1.1)-(1.3), 17 partners did give an answer. As can be seen in Figure 2, more than the half of them (8 out of 17) could give three answers in the positive, but nearly as much of them do not have dedicated calibration staff, no hierarchy and no transparent chain of responsibility (6 out of 17). Three partners do have calibration staff, but lack of somewhat clear structures like hierarchy and/or responsibilities. All in all, it could be said that in case an institute employs calibration staff, most of them also have clear personnel structures.



l na na mana mana

Figure 2 : Analyses of calibration staff features, summarising questions (1.1)-(1.3) of calibration facility questionnaire (part I) among JERICO partners.

In Figure 3a results regarding calibration funding are shown. Of 15 partners only two partners lack funding by the corresponding institute and raise their funding by projects. All the other 13 are funded by the institute itself, 7 of them provide even a constant funding by the institute. 10 out of 13 institutes get their funding by the institute and also by projects. All in all, most partner institutes do have funding for their calibration facility. However, a permanent and reliable calibration system can only maintained by constant budgetary and project funding. This is only affordable for a minority of institutes.

Question (2.4) is analysed separately. We wanted to know if funding is available for upgrading instrumentation and for purchase of new instruments. As Figure 3b is showing, a majority of institutes do have funding for these issues. There is no real dependence on the previous questions (2.1)-(2.3) recognizable.







Figure 3 : (a) Questionnaire analyses regarding calibration funding. Questions (2.1)-(2.3) of part I have been used. (b) Question (2.4) of calibration questionnaire (part I): *Is there separate funding for upgrading or acquiring new instrumentation, etc.?*

Table 1 : Budget for calibration facility	issues of JERICO partner institutes.
---	--------------------------------------

Institute OG	GS	AZTI	CNRS	CSIC	PUERTOS	IFREMER	NOCS	HCMR	HZG	NIVA
Budget in € 27,	,500	Variable	5,000 + <i>x</i>	4,000	15,000	20,000	30,000£	50,000	50,000	25,000- 40,000

The participants of the survey have been asked for an estimate of the annual operating budget for the calibration facility. In Table 1 an overview is presented for 9 institutes which replied to that issue. However, there is no clear view of the budget possible as the numbers vary significantly. NIVA and HZG stress that the total amount depends strongly on the operating platform and the logistics that are needed for (in case of Ferryboxes, the distance to the port is a criterion). HZG declared to account a half-time position plus material costs

Deliverable 4.1- date:22/01/2013



into the total costs of calibration. Generally, it could be supposed that even personnel costs may vary significantly among the project partners.

Questions (3) to (12) examine several general aspects of sensor calibration like quality standards, accreditation, personnel training, quality manuals and control charts.

In a first step, out of these questions, 3 groups have been formed:

- Quality standards (question (3)-(5))
- Documentation (question (6)-(9))
- Collaboration (question (11)+(13))



Figure 4 : Survey of quality standards of calibration facilities in JERICO.

In terms of quality standards, Figure 4 shows answers of partner institutes to quality management standards, accreditation and continual training. A majority of institutes apply quality management standards (9 out of 16) but most of them lack accreditation and continual training (6 out of 16). Below (e.g. in Table 2 and Table 3), details to quality management standards and to continual training are presented.





Figure 5 : Survey of documentation of calibration facilities in JERICO.

Four questions are condensed in the topic documentation and shown in Figure 5. Obviously, nearly all responding JERICO partners can assure an effective traceability chain to primary standards and also nearly all of them use formal quality manuals. However, only a minority (7

C

out of 16) use also control charts (whatever kind they are) and only 8 out of 16 maintain an in-House quality assurance program. So a certain level of documentation has been achieved by most institutes but there is room for improvement for higher quality of documentation.



Figure 6 : Survey of collaboration of calibration facilities in JERICO.

The third group is headed as collaboration of calibration facilities and encloses links to National Metrology Institutes and calibration service for other institutes. It gets clear in Figure 6 that in this group improvement is needed as only 5 JERICO partners could answer in the positive. Both questions are evaluated in detail later on.

In the next step, we summarized all the questions (3) to (12) by counting how often they are answered in the positive. The results are shown in Figure 7. The reliability has a range of 0 to 9, as 9 answers in total could be answered in the positive. The institute's reliability is somewhat sampled in three groups in the figure: 5 institutes only answered in the positive once or never. The middle of the field is represented by 7 institutes with 4-6 answers "yes". 3



out of 17 institutes actually answered in the positive 9 times, so they are supposed to have best reliability.



Figure 7 : Distribution of reliability of JERICO partner institutes to general calibration techniques for questions 3-11 of calibration questionnaire part I.

In the next paragraph, we are going into detail about some issues of general aspects of calibration facilities:

- Question (3): Which Quality Management Standards have been employed to the calibration system?
- Question (5): Does your facility actively endorse a policy of continual training/education of?
- Question (11): Does your facility maintain links of any kind with the National Metrology (NMI)?
- Question (13): Does your facility provide a calibration service for sensors of other (research) institutes?





Partner institute	Quality Management Standards
BSH	ISO 9000
CEFAS	ISO 9001
CNRS	ISO 9000:2000 (OSIL for nutrients, salinity and O2)
HZG	ISO 9000:2000 (Only for Chlorophyll-a)
IFREMER	ISO/IEC 17025
IH	ISO 9000:2000, ISO 10012, ISO/IEC 17025
NOCS	ISO 9000:2000 (All procedures are recorded and calibrations applied to data are traceable and logged as part of the meta data.)
SMHI	ISO 9001, ISO17025
NIVA	ISO9001:2008, accreditation ISO/IEC 17025 and ISO/IEC 17043 for laboratory, registered in Achilles and Sellicha

Table 2 : Quality Management Standards being applied by JERICO partner institutes.

Question (3) asks for the Quality Management Standards employed to the calibration system at the partner institutes. If they could affirm the questions, details should be declared. In total, 8 institutes apply the common quality management standards to their calibration system. The basic requirements for quality management are specified in the ISO 9000 family of standards. ISO 9001 describes the demands to an organization to demonstrate its ability to consistently provide products that meet applicable statutory and regulatory requirements (ISO, 2009).In Table 2 an overview of the used Quality Management Standards is presented. Nearly all institutes apply the quality management standard ISO 9000 / 9001. Three institutes (IFREMER, IH and SMHI) declare to apply also the standard ISO/IEC 17025 which is adopted for accreditation of calibration facilities. In the questionnaire, application of Good Laboratory Practice (GLP) was inquired, only NIVA did mention that it it used at their laboratory. GLP is a quality system concerned with the organisational processing process

C

and conditions, under which non-clinical health and environmental safety studies are planned, performed, monitored, recorded, archived and reported (OECD, 2003). NIVA is also registered in Achilles and Sellicha. Achilles is a cooperation between Norwegian and Danish oil companies and main contractors using the system for getting overview of potential vendors, whereas Sellicha is a cooperation system of energy companies in the Nordic countries.

Partner institute	Calibration training techniques				
HCMR	Participation in international workshops and calibration experiments. Organizing and performing calibration experiments with national partners and associates (e.g University of Aegean, Technical Institute of Athens)				
CEFAS	There is internal training provided for chemistry staff working on nutrient samples at the institute. Central record of the training received by each staff member. A record is kept of the staff analysing each sample including those for quality checks such as Quasimeme. Most of the methods include internal QA standard checks as part of the procedure.				
IFREMER	Metrology (physical and chemical parameter) Statistics				
SMHI	 All personnel that work with analysis, in situ measurements etc. must have a "driving license" for the specific moment. All personnel regularly join training courses concerning the different moments (collecting water samples, analysis, measurements etc). 				
IH	Training for calibration, quality and metrology area attended at OSIL, UK				



The training of calibration staff is supposed to be an important factor for improving calibration quality and the dissemination of calibration techniques. 5 partner institutes declared to train their calibration staff (see Table 3). The methods range from internal training (in terms of metrology, statistics, calibration routines) to participation in workshops at metrology institutions. As less than one third of all partners declare to provide training to their staff, it seems to be an issue for improvement of the general calibration system.

Partner Institutes	Links to NMIs
IFREMER	The calibration laboratory is involved in EMRP project ENV05: Ocean measurement (lead by the PTB: German NMI). The ENV05 project deals with salinity, oxygen, pH, speed of current measurements.
	We participate to Inter laboratory comparisons organised by the French NMI (LNE).
	All the devices are calibrated by the French NMI (LNE)
SMHI	 -Some reference instruments are regularly sent to the Technical Research Institute of Sweden for calibration. -Some sensors however are regularly sent back to the manufacturer for general check-up and calibration.
SYKE	Annual FINAS (Finnish Accreditation Service) accreditation of the main methods.
IH	Client of IPQ (Instituto Português da Qualidade) http://www.bipm.org/en/practical_info/useful_links/nmi.html.
	IPQ calibration services to calibrate the Standard Platinum Resistance Thermometers (SPRT's) satisfying the requirements of the ITS-90, and also a Precision Resistors are used.

Table 4 : Links of JERICO partner institutes to National Metrology Institutes (NMI).



Further on, the links to National Metrology Institutions (NMI) are evaluated (see Table 4). 4 institutes maintain links to a NMI. These links include controls of sensors used by the institutes (e.g. at IFREMER, SMHI, IH) as well as inter laboratory comparisons (IFREMER) and accreditation service (SYKE). IFREMER is even involved in metrology project ENV05 which deals with the improvement of metrological infrastructure required for a reliable monitoring and modelling of ocean processes.

A further general aspect of a calibration system is the service of sensor calibration provided for other institutes. We wanted to know if JERICO partners are offering that service and in case they do so, for which parameters. In Table 5 the results of that survey are shown. Of 13 partner institutes answering this question, only 5 institutes provide a calibration service for other institutes or institutions. IFREMER, IH and OGS provide calibration service for temperature and conductivity sensors. IFREMER provides the service also for a variety of other sensors (pressure, ocean current, pH, turbidity, fluorescence and dissolved oxygen). SYKE has specialized for chlorophyll fluorometer sensors. They stress that this service, however, is only available for project partners of Algaline and thus somewhat limited. Same could be said for NIVA. They provide (turbidity) standards and are also involved in TSM validation for other institutes.





Table 5 : Calibration service provided by JERICO partners for other institutes. Inputs marked with an asterisk mean limited calibration service (e.g. only for project partners (SYKE) or provision of standards (NIVA).

Institute	Does your calibration facility provide a calibration service for sensors of other (research) institutes?	parameters
AZTI	No	-
BSH	No	-
CEFAS	No	-
CSIC	No	-
HCMR	No	-
HZG	No	-
IFREMER	Yes	T, cond, p, ocean current, pH, turb, flu, DO
IH	Yes	T, cond, p
MI	No	-
NIVA	Yes (*)	turb, Chl-a
OGS	Yes	T, cond
PUERTOS	No	-
SYKE	Yes (*)	Chl /Flu

To summarize our findings, we evaluated the reliability of JERICO partner institutes regarding general aspects of calibration. So we analysed the total first part of the questionnaire, summing up questions (1) to (11). It could be said that few partner institutes (2 out of 17) possess an overall good infrastructure for calibration matters.

They employ staff only responsible for calibration and they also have funding from institute budgets as well as from projects. Moreover, they also use and apply all other calibration features mentioned in our questionnaire.



The majority of institutes could answer in the positive for most questions in our questionnaire, but they lack in some points. The vast majority of institutes do not have some kind of accreditation and also no links to national metrology authorities. Most also do not train continually their personnel. But still, these JERICO partners reach a level of reliability between 8 – 11 points.

Some institutes reach only a level of reliability of 6 or below. In several cases they are able to fund their calibration facility but lack of personnel which is employed only for calibration matters.

4.2. Calibration Facilities for Physical Sensors

Physical sensors are the ones with the longer history in the oceanography with significant advancements in the last decade. Following the overall objectives of WP4, the present section will be constituted by the documentation and assessment of existing calibration methodologies, equipment, and reference material currently in use for main parameters to arrive at a consensus on methodologies and specifications. The focus of this section will be on temperature and salinity.

The next step, however, will be the sharing of calibration facilities including joint meetings for documentation of existing calibration infrastructures within JERICO and the identification of potential trans-network "nodes" for these services. So this report will serve as a preparation for these actions as well as for the dissemination of know-how and the fostering of technical collaborations and partnerships to deal with calibration issues for different parameters. This activity will provide material to WP6.

4.2.1. Temperature

For calibration issues of temperature sensors, 10 partner institutes filled in the questionnaire. We added up the reliability points as described above for part II of the questionnaire. Half of all institutes reach a high reliability of 5 out of 6 points. For all relevant questions the majority answered in the positive, except for (8, field calibration) and (9.2, independent quality audits). Only 4 institutes deal with field calibrations of temperature sensors. Question (9.2) asks for independent quality audits to monitor and assess the calibration system for the specified parameter. Also only 4 institutes perform these audits. The highest agreement among the involved partners is achieved for question (4). 9 out of 10 partners declared that their facility ensures an effective traceability chain for temperature calibration.

C

Two questions have been analysed in more detail. In Question (1) we wanted to know, how often the presently used temperature sensor system is calibrated. Routine calibration is overall agreed to be done every 6 or 12 months. Some partners calibrate their sensors before and/or after deployment. It was once indicated that calibration procedures are performed after sensor malfunction. Some partners do an additional calibration check with the help of in-situ bottle samples.

Question (2) asks for a brief description of the calibration setup (see Table 17).

Most institutes use a temperature controlled calibration bath for temperature sensors. As a reference system, platinum resistance thermometers (PRT) are widely used (e.g. Deep Ocean Standard Thermometer SBE 35). Some partners indicate that their reference sensor itself is sent to a national calibration facility once a year or every two years.

	Questions (temperature)	Yes	No
4	Effective traceability chain ensured?	9	1
6	Manual of calibration methods?	8	2
8	Field calibration?	4	7
9.1	Internal quality audits?	7	3
9.2	Independent quality audits?	4	6
10	Archive of issued calibration reports/certificates	8	2

Institute	1. How often do you calibrate the sensor/s or sensor system/s <u>you are</u> presently using for the specified parameter/measurand?
BSH	Once a year
HCMR	6 months
CEFAS	12 months
CNR	Three of the sensors were installed in 2008, one has been installed one week ago after the lost of the previous one. They were calibrated before being mounted on the structure and will be checked against regular CTD casts during cruises.
CNRS	We use an AutoSalinometer (model 8400B) and seawater OSIL standards (one per day during analysis).
HZG	Irregularly, regularly check by bottle samples
IFREMER	Depend on scientists and applicatons: mainly before deployment but some scientists also ask for an after deployment calibration.
NOCS	Annual before and after deployment
OGS	Once every 6 months; Prior to deployment or following a malfunction, always.
SMHI	Once a year
SYKE	Annual check, and re-calibration if needed. In addition weekly / bi-weekly validation with in situ samples.
IH	Before a deployment or once a year.

Table 7 : Calibration interval for temperature sensors (Question 1/part II).

in minini ni ni ni

4.2.2. Salinity

Besides the calibration of temperature sensors, 11 partner institutes also filled in the questionnaire for salinity sensors. The questionnaire scheme is the same as for temperature calibration. So again, reliability up to 6 points could be achieved. Nearly the half of the institutes reaches a good result accounting to 5 points, whereas the other half range between 0 and 4 points. In Table 6 it can be seen that nearly all partners can ensure an effective traceability chain of their calibration scheme. The majority also maintain manuals about calibration methods and procedures and store reports and certificates in archives. However, there seems to be highest potential for improving in doing internal and independent quality audits. Only 3 institutes declared to apply quality audits to their calibration routine.

Going into detail, the calibration interval is comparable to temperature sensors, accounting to 6 to 12 months (see Table 9). Some institutes calibrate before and after deployment, some irregularly. Also recalibration after malfunction was reported. The routine calibration is done by salinometers (common used equipment: Autosal Guildline 8400B) in a calibration bath (Hart 7052), or with bottle samples with IAPSO standard sea water probes from OSIL. Details can be found in Annexes in Table 19.

	Questions (salinity)	Yes	No
4	Effective traceability chain ensured?	9	2
6	Manual of calibration methods?	7	4
8	Field calibration?	6	6
9.1	Internal quality audits?	3	6
9.2	Independent quality audits?	3	7
10	Archive of issued calibration reports/certificates	7	4

Table 8 : Overview of questions for reliability evaluation of salinity sensor calibration.





Institute	1. How often do you calibrate the sensor/s or sensor system/s <u>you are presently using</u> for the specified parameter/measurand?
BSH	Once a year
HCMR	6 months
CEFAS	At start and end of deployment
CNRS	We use an AutoSalinometer (model 8400B) and seawater OSIL standards (one per day during analysis).
HZG	Irregularly, regularly check by bottle samples
IFREMER	Mainly before deployment but some scientists also ask for an after deployment calibration.
NOCS	Monthly during deployment
OGS	Once every 6 months;
	Prior to deployment or following a malfunction, always.
IH	Before a deployment or once a year.

Table 9 : Calibration intervals for salinity/conductivity sensors (Question 1/part II).

4.3. Calibration facilities for Optical Sensors

Optical sensors are used to estimate the chlorophyll of phytoplankton (Chl-a/Flu), the amount of suspended particles (turbidity) or the dissolved oxygen in the water (DO). Measurements are based on fluorescence in the case of chlorophyll-a and light attenuation or scattering in the case of suspended material or dissolved oxygen. Chlorophyll-a fluorescence is widely used as a proxy for total phytoplankton biomass. Two important issues are that chl a-fluorescence may vary due to the composition of phytoplankton communities and that the fluorescence of phytoplankton varies due to photo quenching. Thus irradiation (PAR) is an important parameter in this context but it is also important when primary production is estimated.

For the evaluation of existing facilities for optical sensor calibration, 9 JERICO partners filled in the questionnaire for optical sensors. 7 out of 9 adopted turbidity sensor calibration for

answering the questions. Nearly all institutes have a reliability of 3-4 points out of 6. Two institutes did not answer all questions regarding optical sensors and they also did not answer in the positive.

As for physical sensors, nearly all institutes declare to ensure an effective traceability chain for the specified parameter (5 out of 6 institutes, see Table 10). Most institutes also agree to do field calibration for turbidity sensors and the majority also archive their calibration reports and certificates. There is also agreement to physical sensors about room for improvement. Most institutes do not perform internal and independent quality audits for optical sensors, that is, turbidity sensors.

As for physical sensors, we asked for details concerning the calibration /validation interval for optical sensors and the calibration setup.

The answers are not entirely consistent for turbidity sensors. Some institutes declare a calibration interval of 6-12 months, others calibrate weekly, but only during projects. It is also mentioned that the calibration interval depends on the used instrument. A Turner instrument is calibrated every 6 months, whereas a Seapoint instrument at every deployment. Chlorophyll-a sensor calibration is done every 2-4 weeks, nitrate sensors at every deployment. Some facilities (e.g. HZG, HCMR, CNRS, NOCS) calibrate via a solid formazine standard for checking the drift of the instrument (provided by NIVA). IFREMER uses a calibration bath for measuring the stability in terms of temperature and O_2 . CNRS accounts the costs for an external annual maintenance by the device company to 2000 \in .



Table 10 : Overview of questions for reliability evaluation of turbidity sensor calibration.

	Questions (turbidity)	Yes	No
4	Effective traceability chain ensured?	5	1
6	Manual of calibration methods?	4	2
8	Field calibration?	4	3
9.1	Internal quality audits?	1	5
9.2	Independent quality audits?	0	6
10	Archive of issued calibration reports/certificates	4	1

Table 11 : Calibration intervals for optical se	nsors, e.g. turbidity (turb) (Question1/part II).

Institute	1. How often do you calibrate the sensor/s or sensor system/s <u>you are presently using</u> for the specified parameter/measurand?
HCMR	Turb: The turbidity sensors of the HCMR are calibrated twice a year (6 month interval).
CEFAS	Chl-a: Turner 10AU - 6 months Turb: Seapoint SCF – every deployment
HZG	Chl-a: Every 2-4 weeks
IFREMER	DO: Winkler sampling and titration for reference measurement.Temperature regulated bath that can be filled with fresh water or seawater.Bubbling system to reach different concentrations.
NOCS	Turb: We have limited experience with the calibration of turbidity sensors which was gained during the FerryBox project 2002 to 2005. In that project approximately weekly calibration and checks were made on the system that we were operating on the Southampton-Isle of Wight ferry in 2004.
SYKE	Turb: Annual check and re-calibration if needed. In addition weekly / bi-weekly validation with in situ samples.

Intritutututut

4.4. Calibration facilities for Chemical Sensors

Chemical sensors measuring chemical parameters (e.g. NH_4 , NO_2 , NO_3 , SiO4 and o-PO₄) need frequent calibrations and validation with in-situ samples in order to have a satisfactory quality. This is due to deterioration of chemicals, interference with other substances in the water (seasonal or spatial) and other factors.

The proposed methodology for best practices adopted to chemical sensors includes: the standardisation of the Standard Operation Procedures (SOPs) for calibration, adopting common procedures on comparisons with samples (time interval, sampling procedure etc.). Moreover, practices on analytical methods applied on seawater samples for in-situ validation and long term performances evaluation of chemical sensors (pH, Total Alkalinity, TCO2, dissolved inorganic nutrients, total/dissolved organic carbon) will be documented and harmonized.

In a further step in JERICO, sharing of facilities and inter-calibration exercise between the involved institutions will be performed in order to assure a common lab quality.

Thus, information of existing calibration systems for chemical sensors is needed and has been gathered through the answers given in the questionnaire. However, for our analyses of chemical sensor calibration less information was provided by JERICO partners than for physical and optical sensors. Only 6 institutes did fill in the questionnaire for that part. Moreover, the questionnaires have been filled in for different parameters, so analyses are somewhat more difficult than for physical and optical sensors. An overview of the different parameters is given in Table 12.



Table 12 : Measurand described for chemical sensor calibration by JERICO partners.

Institute	Chemical sensor types
CEFAS	NO ₃
CNRS	NO ₃
HZG	NO _x , NH ₄ , o-PO ₄ , SiO ₂
IFREMER	NH ₄ , NO _x
BSH	рН

Thus, in the following paragraph we concentrate on chemical sensor calibration, not on different parameters itself. All partners show satisfactory results as reliability reaches between 3 and 6 points among them.

Table 13 : Overview of	f questions for	r reliability	evaluation of	chemical	sensor calibration.

	Questions	Yes	No
4	Effective traceability chain ensured?	5	0
6	Manual of calibration methods?	4	1
8	Field calibration?	4	1
9.1	Internal quality audits?	3	2
9.2	Independent quality audits?	1	4
10	Archive of issued calibration reports/certificates	4	1

In Table 13 it turns out that the greatest deficit lays on the realization of independent quality audits, as only 1 out of 5 institutes do so. On the other hand, most partners declare to ensure an effective traceability chain for the respective parameters. Most of them also do field calibration and maintain manuals of calibration methods and procedures.

Deliverable 4.1- date:22/01/2013



Regarding the calibration interval of chemical sensors, it can be seen in Table 14, that because of different parameters, no clear statement can be made. Roughly same calibration interval as for optical or physical sensor is applied, ranging from at each deployment to a constant interval of 6 months. As for optical instruments, mainly standard solutions of different kind are used among the partner institutes. IFREMER is engaged in an intercomparison between several institutes every two years. A conventionally accepted reference solution is applied.

Institute	1. How often do you calibrate the sensor/s or sensor system/s <u>you</u> <u>are presently using</u> for the specified parameter/measurand? Please list the typical calibration interval/s you are employing; note that if you are calibrating irregularly, kindly specify why and when (e.g. before a deployment, following a malfunction, etc.)
BSH	pH : The two point calibration (pH =7; pH=9) is used at the beginning of a measurement series or after changing electrodes
HCMR	FI/ChI-a : The ChI-a sensors of the HCMR are calibrated twice a year (6 month interval).
CNRS	Nitrate : We use AA3 autoanalyzer from Bran Luebbe to calibrate nitrate concentrations from optical sensor. Prior to the deployment of gliders and Argo floats equipped with nitrate sensor, the sensor (ISUS and SUNA) is calibrated in lab using nitrate standard and artificial seawater. 5 to 6 batches are performed with different nitrate concentrations (from 0.5 to 15-20 μ M). This calibration is used to check the "zero", the detection limits and the accuracy of the sensor measurements. Such lab calibration is done for each new deployment. As well, during the first profile, seawater is collected onboard and used to measure nitrate concentrations and check the nitrate values from the sensor transmitted in real-time.
CEFAS	Total oxidised nitrogen: Each deployment
HZG	NOx, NH4, o-PO4, SiO2: 2 – 4 weeks
lfremer	pH/Ammonia : Depend on scientists and applications but mainly before and after deployment.

Table 14 : Calibration intervals for chemical sensors (Question1/part II).

linter for the forther

5. Conclusions

This report is a contribution to task 4.1 of work package 4 of EU project JERICO. The objective of work package 4 is to improve the performance of JERICO observatories and the overall quality of products which are delivered by project partners. The first steps consist on a survey of the existing calibration facilities amongst JERICO partners to evaluate common practises depending on measuring platforms, financial and personnel possibilities. Differences between the facilities are outlined and discussed as well as possible future steps.

In our analyses of the questionnaires which have been distributed among the JERICO partners, we concentrate on general aspects of calibration issues as well as on specifications of different sensor types, physical, optical and chemical sensors to be exact. The questionnaire has been completed by 18 JERICO partners. However, the form has not been filled in entirely by all participants of the survey.

The questionnaire has been divided into two parts. The first one deals with general calibration matters, so the overall constitution of calibration facilities can be evaluated. The main aspects are

- Calibration staff
- Funding for calibration
- General guidelines for operation of calibration facilities (quality management, accreditation, quality sheets, archives etc.
- Links and collaboration with other institutes

In general, most institutes have some kind of funding (ideally both by institute budget and project funding). However, there are differences in the estimated total amount of budget, ranging roughly from 5,000 to 50,000 €. A majority does have project and budget funding and

Deliverable 4.1- date:22/01/2013
also a majority is able to spend money for upgrading. Though only 4 out of 15 institutes have a constant funding which is supposed to be important as sensor calibration is routine work. So this is thought to be an issue for improvement.

A second important issue is supposed to be that some institutes have no staff responsible only for calibration work, although this is supposed to be needed for reliable routine sensor calibration. However, also at this point a wide spread can be observed, i.e. nearly the half of all questionnaire participants declared to employ dedicated staff with a clear hierarchy and chain of responsibility.

The overall reliability shows a wide spread between institutes which can show only features of general calibration issues and institutes which reach highest reliability by

The second part of our analyses on existing calibration facilities evaluates the calibration routines and system for different sensor types. We distinguish between physical, optical and chemical sensors. The JERICO partners have been asked to fill in for each parameter separately. Most questionnaires have been filled out for physical sensors (12). Fewer contributions have been provided for optical (9) and chemical (6) sensors. The main topics of this part of the questionnaire are details of the calibration routine like calibration interval, used instruments, field calibration, quality audits etc.

Calibration routines differ strongly depending on the measuring platform. Thus, instruments are often calibrated before deployment when installed e.g. on a glider. Other instruments offer the possibility to be calibrated more regularly, e.g. every 2-4 weeks. This seems to be the case for Ferrybox systems. So instruments are calibrated in most cases on occasion. Maybe a rethinking should be started to calibrate instrument depending on what is designated originally?



Undoubtedly, a closer cooperation towards harmonisation between calibration facilities is urgently needed as a next step. Considering that calibration costs are a significant part of the regular platform maintenance, it becomes more than evident that scientific operational centres around Europe maintaining calibration facilities must follow the successful example of JERICO, ESONET and EuroSITES:

- Define the calibration community (already done through JERICO)
- Setup a permanent calibration group (this is a JERICO milestone)
- Develop strong links between them (this is in progress through JERICO)
- Start working towards harmonisation of practices (this is in progress through JERICO)
- Seminars like the latest Best practise workshop on Crete in October 2012 can help to close the gap on a more theoretical basis in knowledge and experience between the partner institutes.
- Collaborative trials for basic parameters such as salinity, turbidity and chlorophyll-a (this was done during 8-12 October 2012 at IFREMER calibration lab)
- Adopt common standards as much as possible considering the peculiarities of each infrastructure (this is a JERICO objective)
- Share expertise and facilities (this is successfully done in JERICO)
- Work towards a 2-level system
 - Primary calibration labs. These are accredited labs around Europe able to calibrate secondary reference sensors
 - Secondary calibration labs. These are labs using secondary reference equipment for day-to-day calibration of sensors.

C

During the first 18 months JERICO has significantly advanced on the above through a series of activities, which proved to be particularly successful. Thus, the devoted to calibration sections during the common between WP3 & WP4 workshops in Hamburg, Rome and Palma gave the opportunity to discuss and exchange information on calibration issues across observing platforms. Calibration techniques, problems and challenges for FerryBox, Fixed Platforms and Gliders were thoroughly examined acknowledging commonalities and most importantly differences. Furthermore dedicated workshops to calibration practices for sensor categories (optical, chemical etc) were organized. The first attempt was done on the 9th of February 2012 at SYKE focusing on optical sensors (Chl-a and turbidity). There were 21 attendants and the aims of the workshop where:

- 1. How to perform the primary instrument calibration for fluorometers
 - a. Algae cultures / Solid secondary standards / Chemical standards
 - b. Comparison of instruments
- 2. How to perform validation with field samples
 - a. How to deal with the variable fluorescence yield
- 3. How to prevent bio fouling

The outcomes of the workshop in SYKE, consider optical sensor (Chl-a) calibration to be a 2 level problem starting from the absence of a commonly accepted reference material for Chl-a calibration and the challenges of estimating the Chl-a concentration using fluorescence measurements.

The 1st level problems are the reference materials for Chl-a calibrations:

- Secondary standards:
 - o Best practice to use solid standard to follow instrument performance
 - Traceability of secondary standard (contact manufacturers)
- Chemical standards:
 - Chl-a in acetone (or other solvent) may be solution for some instruments but may not be compatible with others

- Should find better chemical standards for primary calibration
- Are there special problems with instruments working in low range (stability of standards, offset)?

2nd level problem is the conversion from fluorescence to Chl-a concentration. There are many alternatives to estimate Chl-a concentration from fluorescence thus:

- Importance of keeping raw data
- Importance of archiving

Considering the success of the workshop it was decided during the common WP3 & WP4 workshop in Rome to organise a second calibration exercise/workshop at IFREMER focusing on Oxygen, Temperature and Conductivity in an attempt to benefit from the significant experience of IFREMER, which operates one of the few accredited marine calibration labs in EU. As planned it took place in Brest (8-12 October 2012) in parallel with the SeaTechWeek event. The major aim was to compare the calibration methods used by each laboratory by organizing an inter-laboratory comparison. Four partners participated with six different sensors and a report is in preparation.

Additionally workshops like the SeaTechWeek in October 2012 in Brest can lead the right way to a more consistent calibration system throughout JERICO as well as to the outside community interested to set up and operate calibration facilities.



Annexes and References

Annexes

Table 15 : Involved institutes being a partner in JERICO and the contact persons.

Institute	Abbreviation	Contact person
AZTI-Tecnalia	AZTI	Carlos Hernández, Luis Cuesta
Bundesamt für Seeschiffahrt und Hydrographie	BSH	Detlev Machozcek
Centre for Environment, Fisheries and Aquaculture Science	CEFAS	Naomi Greenwood, Dave Sivyer
CONSIGLIO NAZIONALE DELLE RICERCHE	CNR	Roberto Colucci
National Center for Scientific Research	CNRS	Laurent Coppola
AGENCIA ESTATAL CONSEJO SUPERIOR DE INVESTIGACIONES CIENTIFICAS	CSIC	Joaquín Tintoré
HELLENIC CENTRE FOR MARINE RESEARCH	HCMR	George Petihakis, Manolis Ntoumas
Helmholtz Zentrum Geesthacht	HZG	Wilhelm Petersen
Institut français de recherche pour l'exploitation de la mer	IFREMER	Florence Salvetat
INSTITUTO HIDROGRAFICO	IH	Manuel Marreiros
Marine Institute	MI	Glenn Nolan
Norwegian Institute for Water Research	NIVA	Kai Sørensen
National Oceanography Centre Liverpool	NOCL	John Howarth
National Oceanography Centre Southampton	NOCS	Mark Hartman
ISTITUTO NAZIONALE DI OCEANOGRAFIA E DI GEOFISICA SPERIMENTALE	OGS	Rajesh Nair
Puerto del Estado	PUERTOS	Begoña Pérez Gómez, Marta de Alfonso Alonso- Muñoyerro
SVERIGES METEOROLOGISKA OCH HYDROLOGISKA INSTITUT	SMHI	Henrik Lindh, Johan Håkansson, Bengt Karlson
SUOMEN YMPARISTOKESKUS	SYKE	Jukka Seppälä

Deliverable 4.1- date:22/01/2013

lii lii lii lii lii lii lii l

Table 16 : Questionnaire – Part I (General information about calibration facilities). Marked questions have been used for general reliability evaluation.

1.1	Does your calibrating facility possess a well-defined organizational framework with Dedicated staff?	
1.2	Does your calibrating facility possess a well-defined organizational framework with Clear hierarchy?	
1.3	Does your calibrating facility possess a well-defined organizational framework with Transparent chain of responsibility for management, technical/scientific and operational decisions)?	
2.1	Briefly describe the size and nature of the annual operating budget of your facility.	
	Is it funded by your Institute/Centre?	
2.2	If Yes , is the funding constant?	
2.3	Is it funded by Projects?	
2.4	Is there separate funding for upgrading or acquiring new instrumentation, etc.?	
3	Does your facility employ Quality Management Standards - ISO 9000:2000, ISO 10012, Good Laboratory Practice (GLP), and the like - to its calibration systems?	~
4	Does your facility possess any kind of accreditation for the calibrations?	✓
5	Does your facility actively endorse a policy of continual training/education of personnel actively involved in calibration activity?	√
6	Does your facility maintain a documented in-house Quality Assurance Programme?	✓
7	Does your facility maintain a formal Quality Manual (containing, at the very least, listings and descriptions of equipment and procedures, maintenance/calibration records and certificates for instrumentation, and safety precautions and regulations)?	√
8	Does your facility make use of control charts (Shewhart Charts, other) for Quality Control purposes?	~
9	Can your facility assure an effective traceability chain to primary standards or, in their absence, to conventionally accepted reference material (certified or otherwise)?	~
10	Does your facility furnish uncertainty estimations for its calibration systems?	~
11	Does your facility maintain links of any kind with the National Metrology Institute/s (NMI/s) of your country?	√
12	In the list of sensors below, please indicate only the ones that you currently <u>never</u> calibrate yourselves; in each case, kindly report the calibration provider (manufacturer, other) and the typical calibration interval (trimonthly, half-yearly, yearly, other) you are presently employing.	
13	Does your calibration facility provide a calibration service for sensors of other (research) institutes?	

Table 17 : Questionnaire – Part II (Detailed questions answered separately for physical, optical and chemical sensor types). Marked questions have been used for reliability evaluation of different sensor types.

-		
1	How often do you calibrate the sensor/s or sensor system/s <u>you are presently using</u> for the specified parameter/measurand: please list the typical calibration interval/s you are employing; note that if you are calibrating irregularly, kindly specify why and when (e.g. before a deployment, following a malfunction, etc.).	
2	Please provide a brief description of the calibration setup, including a list of the principal equipment, reference material (certified and/or conventionally accepted) and instrumentation involved in a typical calibration operation.	
3	Do you employ reference material which are mutable or unstable (e.g. secondary standards, reagent solutions, gas mixtures, pressure generators, etc.) to calibrate the sensor/s or sensor system/s <u>you are presently using</u> for the specified parameter/measurand.	
4	In your view, does your facility ensure an effective traceability chain for the specified parameter/measurand?	~
5	Please provide a brief description of the procedures employed to ensure adherence of the performances of the principal equipment and reference instrumentation of the calibration setup to factory specifications (in-house monitoring of performance, in loco re-calibration, servicing by the manufacturer, etc.).	
6	Does your facility maintain a Manual with a description of the calibration method and the measuring procedures, together with details of sample treatment and preparation when these steps are present?	√
7	In your view, is regular factory calibration/servicing necessary to obtain optimal performances from your sensors/instrumentation for the specified parameter/measurand in the field?	
8	Do you perform field calibrations for the specified parameter/measurand?	✓
9.1	Does your facility perform:	✓
	internal quality audits to monitor and assess its calibration system for the specified parameter?	
9.2	Does your facility perform:	✓
	independent quality audits to monitor and assess its calibration system for the specified parameter?	
10	Does your facility actively maintain an archive containing issued calibration reports/certificates for the specified parameter/measurand?	~
11	Do you have any suggestions or ideas for improving the quality of your calibrations for any particular sensor/sensor system <u>you are presently using</u> for the specified parameter/measurand (e.g. innovative reference material, modifications to existing methodologies or new methodologies you have developed, etc.)?	
12	Do you have any suggestions or ideas for improving the general quality of the calibration of sensors or instruments for measuring the specified parameter/measurand (e.g. testing and promoting the use of new reference material, development of new methodologies, etc.)?	





Table 18 : Description of calibration routine for temperature sensors (Question 2/part II).

Institute	2. Please provide a brief description of the calibration setup, including a list of the principal equipment, reference material and instrumentation.
BSH	Calibration bath, reference system: Pt-100 reference resistance, triple-point-cell: water, Gallium, precision thermometry bridge CASL, type F18
HCMR	 Baths (1 Seawater Heating Tank -1.100 lt, 1 Sea/Fresh water Temperature Controlled Bath -120 lt) Deep Ocean Standards Thermometer SBE 35 as temperature reference sensor
CEFAS	Temperature controlled bath with calibrated PRTs used to give water temperature. Sensor is calibrated according to manufacturer SOP. PRT are sent to calibration facility every two years.
CNR	Calibration is done using a "mercury well thermometer" (termometro a pozzetto a mercurio) dropped down in a tank with the probe to calibrate.
CNRS	Local calibration with OSIL standards so far
HZG	bottle samples are measured against certified seawater standards
IFREMER	- Bath (1 of oil, 4 of fresh water or seawater (2 of 25 litres, 1 of 800 litres, 1 of 100 litres) , 1 of glycol water)
	- resistor bridge (2 Guildline, 1 Measurement International)
	- standard resistance thermometer (4 Rosemount 162CE and 1 Leeds and Northrup 8167-25)
	- standardresitors (Guildline: 2 of 10 ohms, 1 of 100 ohms and 1 of 1000 ohms)
NOCS	Triple point cell in NOC calibration facility
OGS	Hart 1590 Precision Digital Thermometer with Metal-sheath SPRT
	(Rosemount 162CE / Hart 5699)
	SBE41 CP-OGS Conductivity & Temperature Monitor
	Hart 7312 TPW Maintenance Bath
	Hart 9230 Ga Cell Maintenance Bath
SMHI	A pressure chamber with a "normal" is used for calibrations. The "normal" is sent to a national facility once a year.
SYKE	Algae culture Monoraphidium contortum used as a reference material. Chla concentration of the
	culture determined with standard methods using fluorometry and pure Chla standard (Sigma).
	Algae culture circulated through several fluorometers simultaneously and the calibration
	coefficients adjusted so that all instruments converge. Additional check with solid secondary
	standard is carried out for the Turner Scufa.
IH	Conductivity reference: IAPSO standard sea water from OSIL;
	Salinometer Autosal 8400 B from Guildline with Autosal interface and
	software from OSIL;
	SBE 4 Conductivity sensor and pump from Seabird;
	5 calibration bath with sea water and salinity from 2-40.
	Deliverable 4.1- date:22/01/2013

աստար

Table 19 : Description of calibration routine for salinity/conductivity sensors (Question 2/part	
II).	

Institute	2. Please provide a brief description of the calibration setup, including a list of the principal equipment, reference material and instrumentation.
BSH	Guildline 8400 B
HCMR	 Baths (1 Seawater Heating Tank -1.100 lt, 1 Sea/Fresh water Temperature Controlled Bath -120 lt) AutoSal 8400A Salinometer as salinity reference Deep Ocean Standards Thermometer SBE 35 as temperature reference sensor
CEFAS	Guildline portable salinometer used for analysis of discrete water samples. Instrument is standardised with IAPSO standard seawater.
HZG	bottle samples are measured against certified seawater standards
IFREMER	GuildlineportasalSalinometer
NOCS	Water sample collected during monthly manned crossing
	Measured by Guildline AutoSal salinometer on return to shore based laboratory
	Referenced to Ocean Scientific International Ltd. IAPSO Standard Seawater
	Five minute data record from crossing fitted to data from samples (about 20 per crossing).
	Underway data for year are adjusted on basis of best fit to monthly sample values.
OGS	Hart 7052 Seawater Calibration Bath, Guildline 5010 Seawater Calibration Bath, Hart 1590 Precision Digital Thermometer withMetal-sheath SPRT (Rosemount 162CE / Hart 5699), SBE41 CP-OGS Conductivity & Temperature Monitor,
	Laboratory Salinometer (GuildlineAutosal 8400B)
IH	Conductivity reference: IAPSO standard sea water from OSIL;
	Salinometer Autosal 8400 B from Guildline with Autosal interface and
	software from OSIL;
	SBE 4 Conductivity sensor and pump from Seabird;
	5 calibration bath with sea water and salinity from 2-40.

Deliverable 4.1- date:22/01/2013

հուսուսուսու

(Question 2/part II).

Institute	2. Please provide a brief description of the calibration setup, including a list of the principal equipment, reference material and instrumentation.
HCMR	-Special container
	-Reference solutions of known concentration and size
	- Measurement equipment (lab multi meter etc)
CEFAS	Turner 10AU calibration SOP
CNRS	The AA3 is calibrated every year through a national inter-calibration procedure (IFREMER and CNRS labs). For each nutrients analysis, we used nutrient standards to check the concentrations accuracy. Finally, the device company is doing an annual maintenance procedure to verify the technical accuracy of the instrument (cost 2000€ per year).
HZG	check of the drift of the fluorescence sensor by a solid fluorescence standard, not a real calibration
IFREMER	Bath characterization (homogeneity and stability in temperature and O2)
	Calibration of: titrator, pipette and scales.
NOCS	For general operation in the later years for running our FerryBox system between 2002 and 2010, we did not attempt to calibrate the fluorimeter signal because along the route the relationship between fluorescence and chlorophyll-a varied greatly with respect to position along the track and with time as the dominant plankton species types changed. We used the raw fluorescence data as an indicator of active biological production. The oxygen data was then used to provide an estimate of biological production that is more precise estimate of change in biomass than is Chlorophyll-a estimated from fluorescence.
	During the FerryBox 2002-2005 when we were using a Chelsea Instruments fluorimeter we tested the use of a check standard. This was extracted Chlorophyll-a set in an acrylic resin block. This provided a very stable check on the fluorimeter output and was superior to the test unit Turner supply for testing their cyclops units. We tested one in 2008.
	Turbidity:
	(1) Kai Sorensen, NIVA supplied a Fomazine standard that was used by different groups. Dilutions of this suspension were measured during service visits to the ferry.
	(2) In addition during service visits a set of solid optical filters made of clear plastic containing suspended particles were used to check the stability of the turbidity sensor before and after cleaning.
	(3) We collected in-situ samples that were filtered and weighed to provide data on the suspended sediment load during a set of regular manned crossings on the ferry.
SYKE	Algae culture Monoraphidium contortum used as a reference material. Chl-a concentration of the culture determined with standard methods using fluorometry and pure Chl-a standard (Sigma). Algae culture circulated through several fluorometers simultaneously and the calibration coefficients adjusted so that all instruments converge. Additional check with solid secondary standard is carried out for the Turner Scufa.
	Turbidity: Calibration media made with formazine is circulated through several instruments simultaneously and the calibration coefficients adjusted so that all instruments converge.

Table 21 : Description of calibration routine for various chemical sensors (e.g. pH, FI/Chl-a etc.)

Deliverable 4.1- date:22/01/2013

աստուսու

(Question 2/part II).

Institute	2. Please provide a brief description of your calibration setup for the specified parameter/ measurand, including a list of the principal equipment, reference material (certified and/or conventionally accepted) and instrumentation involved in a typical calibration operation.
BSH	pH : Reference material: Titrisol buffer-concentrate pH 7,00 <u>+</u> 0,02 (Merck 1.09887)
	Titrisol buffer concentrate pH 9,00 <u>+</u> 0,02(Merck 1.09889)
	Reference material are tempered at 20 °C
HCMR	FI/Chl-a:
	-Dark room
	-Special container
	-Reference solutions of known concentration and size
	- Measurement equipment (lab multi meter etc)
CEFAS	Total oxidised nitrogen : It is a wet chemical sensor and employs a standard of known concentration which has been analysed in the lab. Before each deployment a linearity check is carried out to verify linearity and range of each instrument.
CNRS	We used a Metrohm device to measure the dissolved oxygen concentrations by Winkler method. These concentrations are used to calibrate the SBE43 sensor. The comparison between O2 from sensor and O2 from titration give a new Soc coefficient from the Seabird calibration file. This allows us to refit O2 vertical profiles after measurements and correct the O2 sensor drift that could happen over the year. In addition to that, a mechanical calibration is done by Seabird US every year.
HZG	NOx, NH4, o-PO4, SiO2: calibration aboard the ship with one standard
IFREMER	pH: Standard solutions.
	Ammonia : Each instrument is calibrated using home made standard solutions, moreover intercomparison between several laboratories is also performed every 2 years using a conventionally accepted reference solution (IFREMER Home made).



արդարորությու

References

ISO (2009): Selection and use of the ISO 9000 family of standards. <u>http://www.iso.org/iso/iso 9000 selection and use-2009.pdf</u>, ISBN 978-92-67-10494-2

OECD (2003): OECD Principles on Good Laboratory Practice. OECD Series on Principles of Good Laboratory Practice and Compliance Monitoring, No. 1, OECD Publishing. Doi: 10.1787/9789264078536-en