Configuration and calibration handbook for new RECOPESCA sensors

Work Package 7 – Deliverable 7.5

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THE OCEAN OF TOMORROW

COOPERATION

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Deliverable 7.5 – Configuration and calibration handbook for new RECOPESCA sensors

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Objectives
Establish functional specifications based on previous experience and in view of NeXOS' objectives of robustness and efficiency

Description of Work
Task 7.2 Optimisation and calibration of the prototypes. Leader: nke; Duration: M22-M39 This work package will describe validation and calibration the the sensors in metrological laboratories and later test them *in situ*. The new sensors for oxygen, fluorescence (as a proxy of chlorophyll) will undergo a series of validation tests to ascertain the accuracy, drift and response time of each probe. The testing will be conducted in the laboratories of nke and IFREMER so results can be compared. Following these qualification tests, the calibration procedures will also be considered and a report written for each sensor. The calibration procedures must be such that implementation by any metrological laboratory is possible. IFREMER will perform the first *in situ* tests on a known fishing vessel in case modifications are required. CNR-ISMAR will then test the new prototypes during oceanographic and fishing cruises, and compare them with commercial sensors by parallel profiling of the water column. To recover the data from the vessels RECOPESCA data concentrators will be implemented. A software layer will be added (WP4) to warrant interoperability with international data portals through Sensor Web Enablement. Full system integration and validation will be performed in WP8 and demonstrated in WP9.

Input needed
▪ D1.3: Project implementation plan

Task work plan
D7.5. **Configuration and calibration handbook for new RECOPESCA sensors** This deliverable will justify part of the work done in Task 7.2 regarding the configuration and calibration of new RECOPESCA sensors. The final objective of the WP7 is to produce inexpensive, but accurate sensors that are sufficiently robust to be used on fishing gears. In this task, we first specify and then develop two new EAF probes to measure oxygen and chlorophyll and compatible with the RECOPESCA system. The oxygen and chlorophyll sensors were first put through mechanical and environmental tests on the test benches at the Ifremer RTD facilities to validate the required AFNOR and NF X10-812 standards, and then underwent

metrological validation at the Ifremer RDT laboratories.

Ifremer, with the collaboration of CNR ISMAR, performed preliminary tests at sea starting in November 2016. The tests will be done on board the research vessel *G. Dellaporta* with the two prototypes. All the functionalities of the EAF probes and the whole system with an updated concentrator were tested.

Three other STPO2 and STPFluo probes are under production by nke. They will be available in early January.

When this task is completed, the sea trials will begin in January 2017 in Norway, Italy and France.

Detailed schedule:

- Oct 16: CNR & REC send concentrators to NKE for upgrade
- Oct 16: IFREMER completes tests (vibrations and shocks) on prototype sensors
- 2-13 Nov 16: Test/validation of prototype sensors on board a research vessel (CNR & IFREMER)
- 17-21 Dec 16: Sensor calibration vs water samples (analysis completed in Feb 17)
- Jan 17: Integration of new sensors in Italy, Norway and France
- Jan-Mar 17: Validation (sensors, system, data)
- Apr-Aug 17: Demonstration until the final dissemination workshop.

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1 Introduction

The first step of Task 7.2 was to design and build new prototypes of optical sensors for integration in OEM (Original Equipment Manufacturer) detection cells to measure two physicochemical parameters, dissolved oxygen and chlorophyll *a*. These prototypes are intended for deployment from fishing vessels during measurement campaigns and for interfacing with the RECOPESCA network architecture.

The sensors, designed to be robust and accurate, were constructed to detect dissolved oxygen and fluorescence concentrations in coastal waters at depths ranging from 0 to 300 meters. The EAF (ecosystem approach to fisheries) prototypes are compliant with the existing RECOPESCA architecture.

Nke developed the prototype sensors EAF STPO_2 and EAF STPFluo. They were made available for testing in May 2016.

Metrological assessment and environmental testing were carried out by Ifremer in collaboration with nke. Test plans were written by nke and Ifremer in accordance with AFNOR and NF X10-812 standards for mechanical strength, climate exposure and salt spray. The objectives were to ascertain the performance of the EAF sensors in terms of uncertainty, drift, linearity, response time and mechanical resistance.

This document is a public guide for the implementation and calibration of the new sensors in compliance with RECOPESCA.

2 Material available

GENERAL PURPOSE OF THE RECOPESCA SYSTEM

The RECOPESCA system is a device intended for installation on fishing vessels. It was initially developed to analyse the extent of the fishing effort.

On the vessel, several electronic devices are installed including:

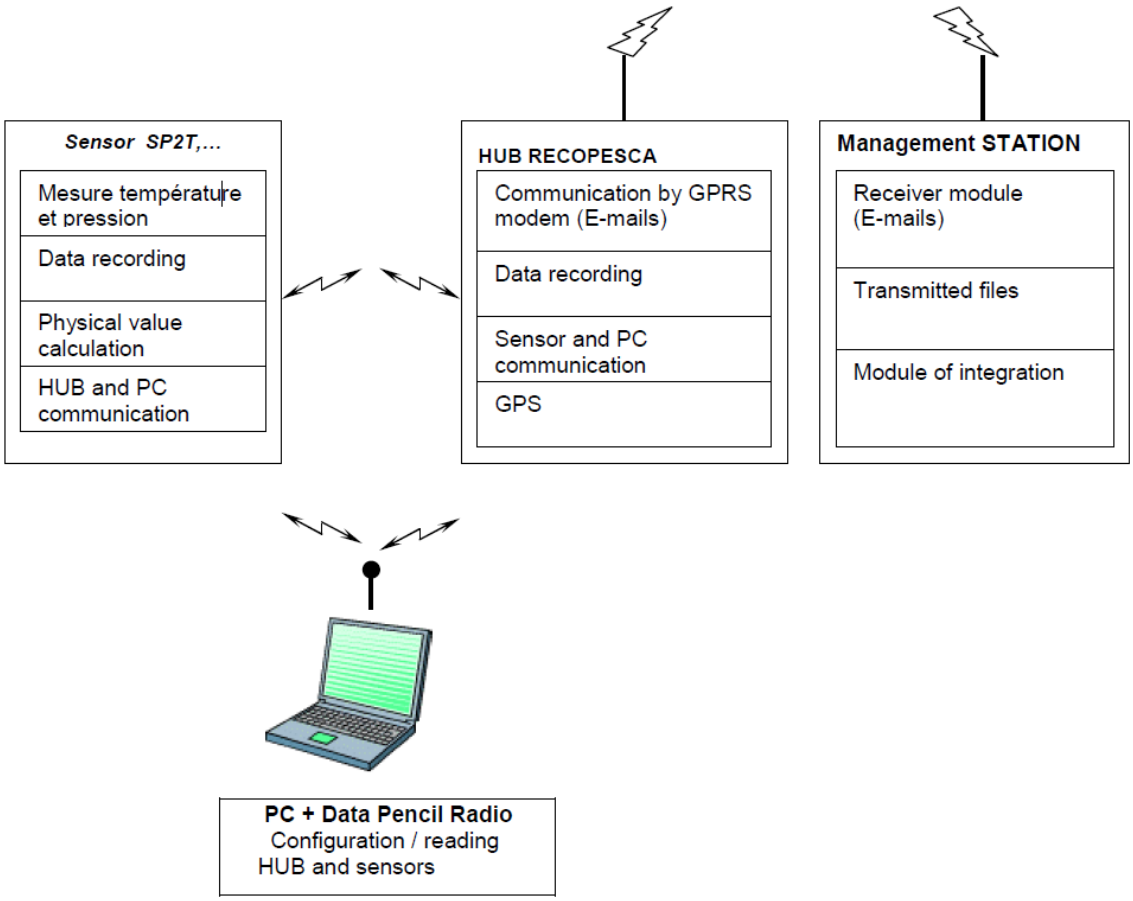
- One or more probes, such as data loggers for temperature and pressure (SP2T), temperature, pressure and salinity (STPS), temperature, pressure and turbidity (STPT) and the two new EAF probes. They are installed on the fishing gears (trawls, lockers, longlines, etc.) to measure the characteristics of the water during fishing operations.

- One or more SLFs: loggers that measure the unreeled length of the fishing gear.
- One concentrator or HUB RECOPESCA: a device that receives and stocks measurement files emitted by the data loggers, it also records the vessel's GPS positions and transmits the files to the management station by GPRS (e-mail).

On shore, a management station receives the e-mails sent by the concentrator.

The RECOPESCA system also includes a Radio Data Pencil connected to a laptop computer and installation software "RECOPESCA.exe". The Radio Data Pencil is a specific component developed by NKE for RECOPESCA to communicate wireless with the sensors. The latter two elements are used to setup the concentrator.

The "WINMEMO2.exe" installation software configures the $STPO_2$ and STPFluo probes.



2.1 Concentrator

The concentrator, installed on board of fishing vessels, includes the following components:

- An electronic board that includes a microprocessor, memory storage, embedded software and a 868 MHz radio transmitter/receiver circuit board,
- A radio GSM/GPRS modem,
- A GPS [updates automatic time of the hub at each valid position (UTC, Coordinated Universal Time)].

The probes transmit their measurement files to the concentrator, using the 868 MHz radio link. The data are temporarily stored in the internal memory of the concentrator before being transmitted via the GPRS modem to the management station. The concentrator also integrates GPS (geodetic positions storage) which allows correlation with the measurement files.

The concentrator resynchronizes the time and date of each data transfer. The time and date transmitted to loggers are in UT (Universal Time) format.

Startup and setup of the concentrator is performed using the Radio Data Pencil that is connected to a laptop and “RECOPESCA.exe” software.

2.2 EAF STPO2 probes

Performances of the EAF STPO2 probes for oxygen measurements:

Sensor	Resolution (Minimum Detection Limit)	Range	Accuracy	Response Time (*)		Maintenance periodicity	Memory capacity
				Stationary	Profiler		1 measurement/minute
Pressure	10 cm	0-300 m	50-100 cm	<3 s at 63%	<0.5 s at 63%	6 months – 1 year	6 months
Temperature	0.01°C	-2 to 35°C	<0.05°C	<3 s at 63%	<0.5 s at 63%	6 months – 1 year	6 months
Oxygen (OEM)	0.02% (0.01 mg/L)	0-100% O_2 (from 0 to 45 mg/L)	(**)	~10 sec		3 months	3 months

(*) That means 63 % of the expected value will be reached after less than 3 or 0,5 seconds

(**) Will be determined at the end of the project according to the results obtained

Response time

Duration between the instant when an input quantity value of a measuring instrument or measuring system is subjected to an abrupt change between two specified constant quantity values and the instant when a corresponding indication settles within specified limits around its final steady value

State of the art for Response time measurement

No norms was found concerning time response measurement protocol for sensors

Definitions can be found.

Sources : http://iamechatronics.com/glossary/Glossary-1/

T63

A measure of device response. It is measured by applying a small (usually 1-5%) step input to the system. T63 is measured from the time the step input is initiated to the time when the system output reaches 63% of the final steady-state value. It is the combined total of the system Dead Time (Td) and the system Time Constant (t). (See Dead Time and Time Constant.)

Dead Time

The time interval (Td) in which no response of the system is detected following a small (usually 0.25% - 5%) step input. It is measured from the time the step input is initiated to the first detectable response of the system being tested. Dead Time can apply to a valve assembly or to the entire process.

Time Constant

A time parameter that normally applies to a first-order element. It is the time interval measured from the first detectable response of the system to a small (usually 0.25% - 5%) step input until the system output reaches 63% of its final steady-state value. (See T63.) When applied to an open-loop process, the time constant is usually designated as τ (Tau). When applied to a closed-loop system, the time constant is usually designated as λ (Lambda).

2.3 EAF STPFluo probes

Performance of the EAF STPFluo probes for fluorescence measurements

Sensor	Resolution (Minimum Detection Limit)	Range	Accuracy	Response Time (*)		Maintenance periodicity	Memory capacity
				Stationary	Profiler		1 measurement/minute
Pressure	10 cm	0-300 m	50-100 cm	<3 s at 63%	<0.5 s at 63%	6 months – 1 year	6 months
Temperature	0.01°C	-2 to 35°C	<0.05°C	<3 s at 63%	<0.5 s at 63%	6 months – 1 year	6 months
Chlorophyll *a* OEM (Fluorescence)	0.025 µg/L	0 to 500 µg/L	(**)			3 months	3 months

(*) That means 63 % of the expected value will be reached after less than 3 or 0,5 seconds

(**) Will be determined at the end of the project according to the results obtained

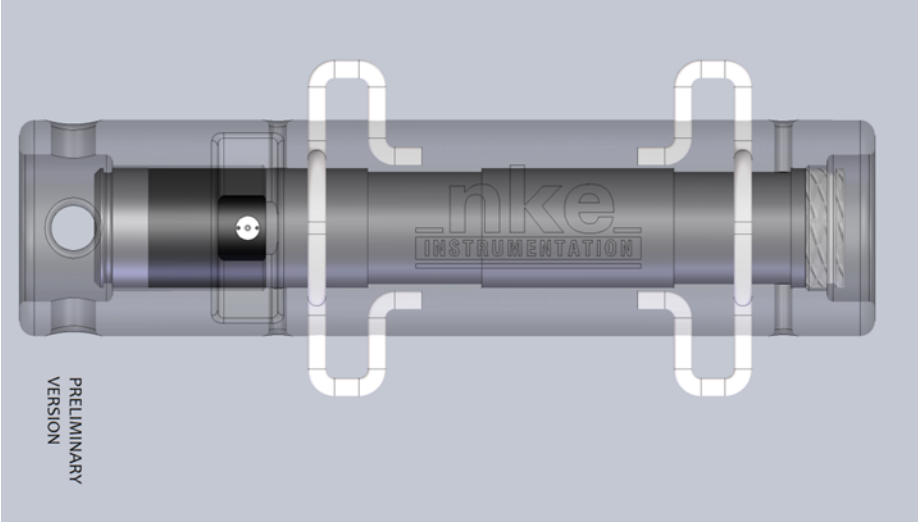
2.4 Hard- and software

- 1 laptop with a Radio Data Pencil for wireless data transfer;
- 1 “WinMemo2” software (latest version) for configuration and post-processing analysis of oxygen and chlorophyll *a* data.

2.5 Sensor protections

The protection avoids damage which may be caused to the sensors by fishing gear. The last design is modify to satisfy the time response of temperature on the additional replicate sensors.

Drawing of the first design:



The sensor is encapsulated in a polyurethane protector in which stainless steel inserts are added. These inserts serve to secure the assembly to the fishing gear. A hole is made in the protection where the temperature sensor is located.

3 Configuration

This chapter is a help for configuring and handling the RECOPESCA concentrator and sensors. The configuration of the sensors is indispensable for use in operation on fishing vessels. This part of the handbook is also useful for the implementation of the sensors in the laboratory during the calibration work.

3.1 Concentrator

3.1.1 Configuration of the concentrator transmission parameters

3.1.1.1 Equipment required

- RECOPESCA concentrator
- GSM cellular phone
- Laptop with operational internet access
- Radio Data Pencil

3.1.1.2 Removal of the PIN code request

Before installing the SIM card in the concentrator modem, the PIN code request must be removed in the following way:

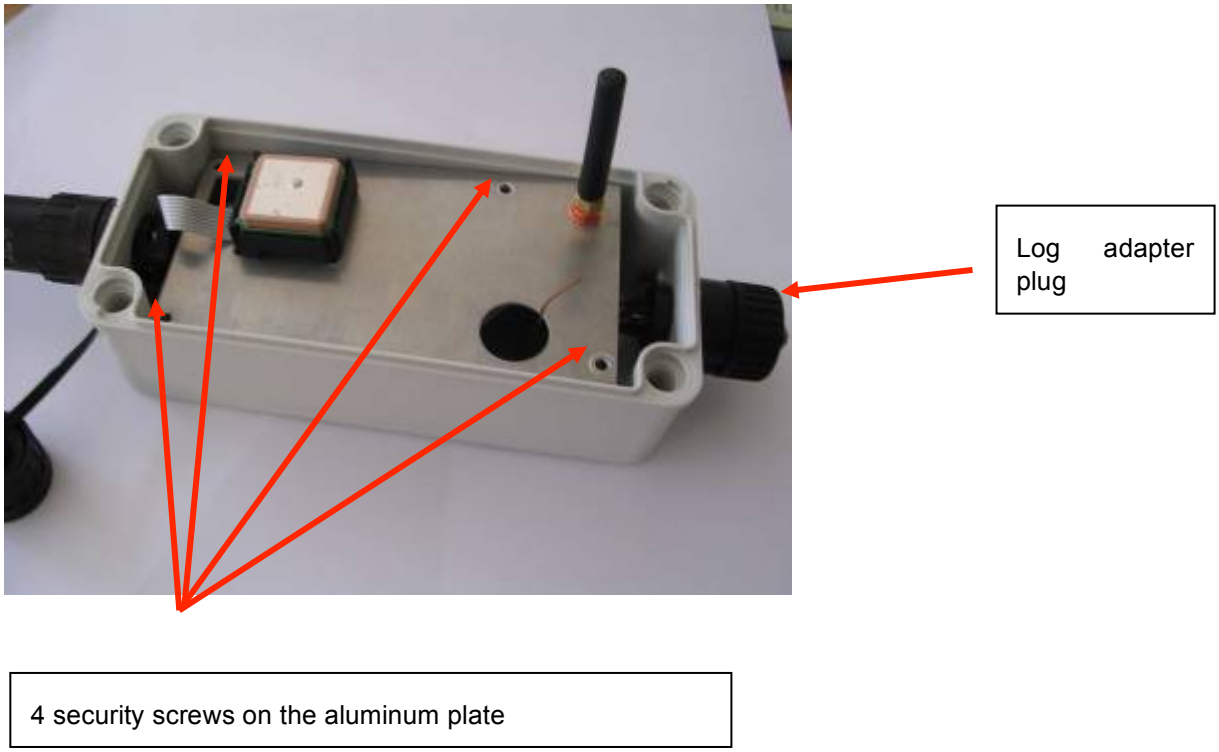
- Insert the SIM card in a cellular phone;
- Turn on the cell phone and enter the PIN code provided by the phone operator;
- Access the settings to deactivate the PIN code;
- Deactivate the PIN code.

3.1.1.3 Creation of an e-mail account

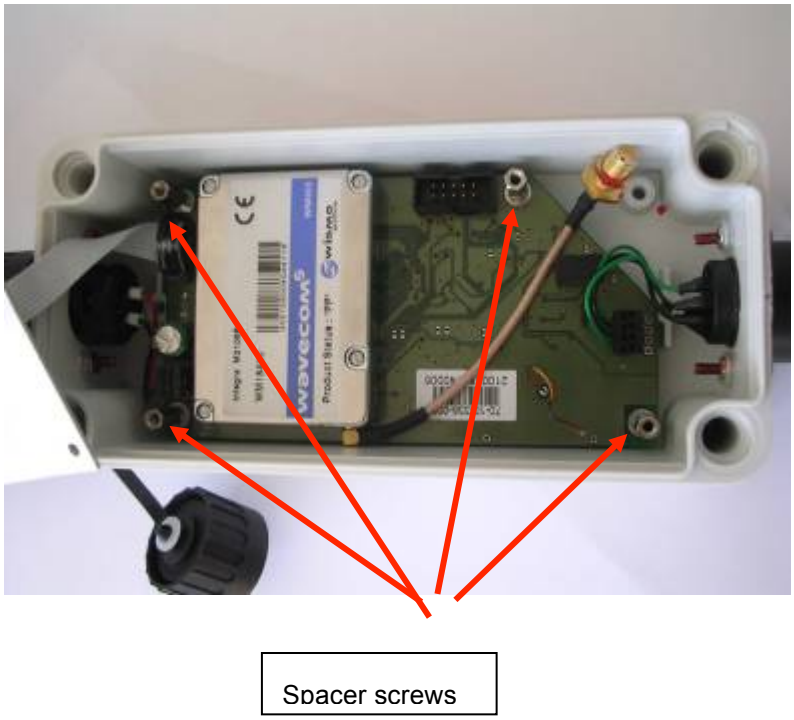
Note the telephone number associated with the SIM card and log on to the Internet site of the phone operator to create an email account associated with the SIM card.

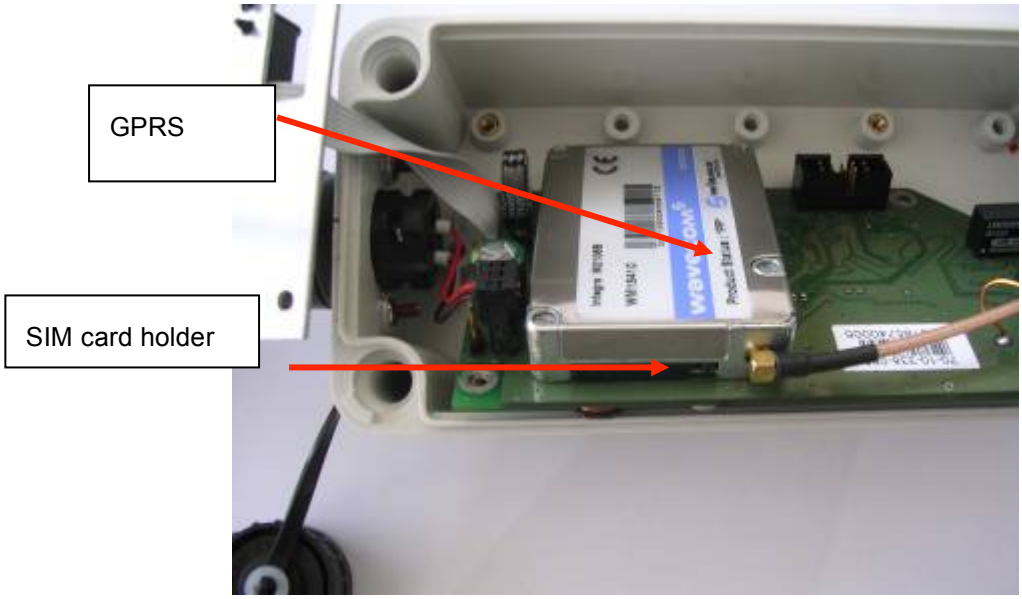
3.1.2 Installation of the SIM card in the modem

- Remove the cover of the concentrator;
- Unscrew the 4 security screws from the aluminium plate;



- Remove the aluminium plate;
- Disconnect the log adapter plug from the electronic board;
- Remove the four spacer screws from the electronic board.





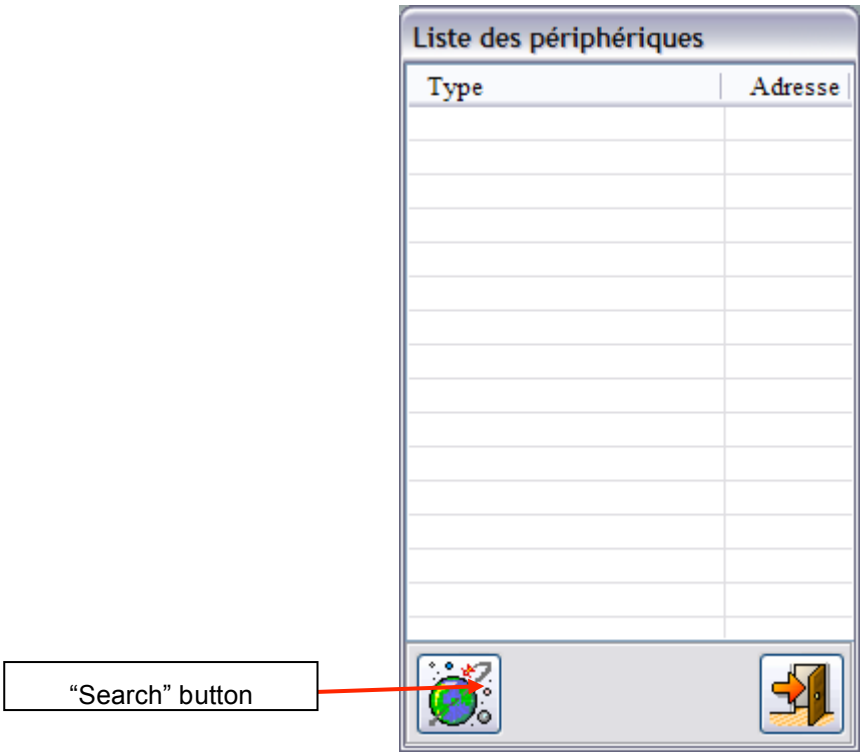
- Remove the SIM card holder from the modem using a stylus;
- Insert the SIM card in the proper position in its slot;
- Put the SIM card holder back in the modem;
- Fasten the 4 spacer screws;
- Plug the log adapter back into the electronic board;
- Fasten the 4 screws on the aluminium plate;
- Screw the cover back on.

3.1.3 Setup of the concentrator

Note: This procedure must be done before installation on the ship.

3.1.3.1 Verification or change of settings

- Plug the concentrator into an electric supply with between 8 and 30 V DC;
- Wait 2 minutes;
- Launch the concentrator.exe software application;
- Click on the “Scan for devices” button;

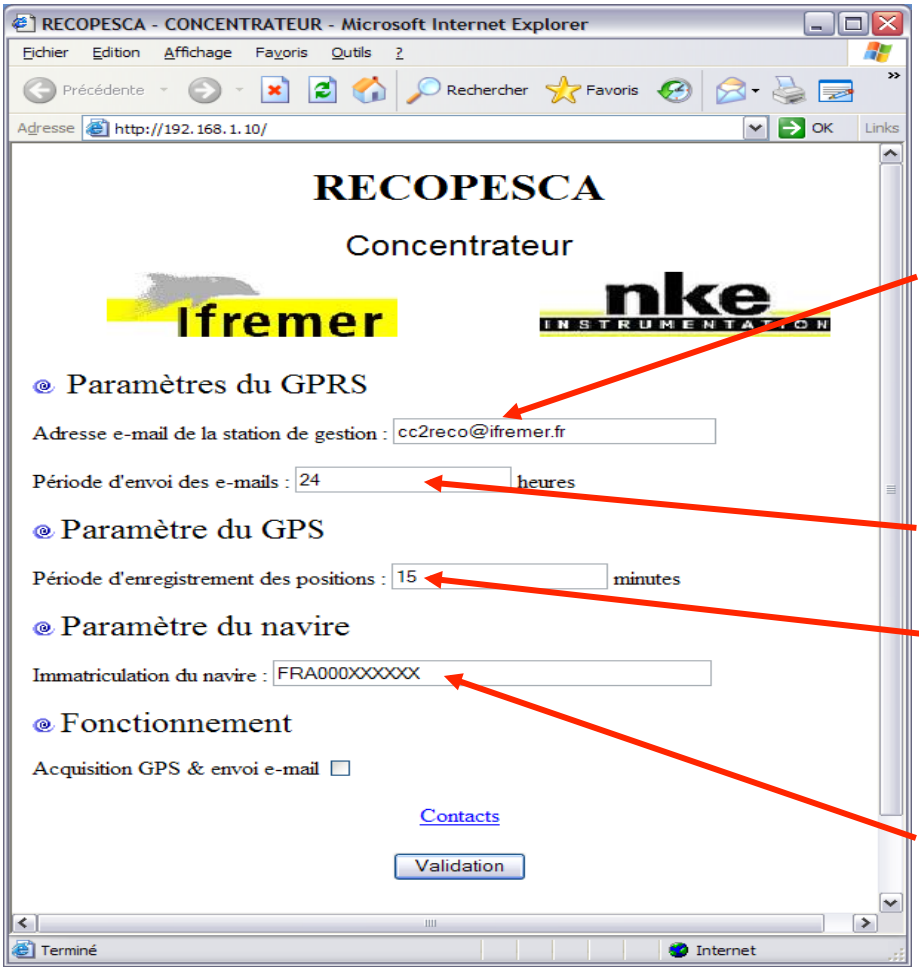


- Click on the “Search” button;

- Double-click on the name of the concentrator to be set up.

Once connected, the “Configuration”, “Téléchargement” and “Dépannage” buttons are activated as shown below;

- Click on the “Configuration HTTP” button for access to the concentrator setup window;
- Enter the following field values according to the example below:



cc2reco@ifremer.fr

Frequency of data transmission to the address above:

Value: **24** hours

15 minutes

Ship registration number.

- Leave the box “Acquisition GPS & envoi e-mail” unticked(*);
- Click on “Validation”;
- After receiving a message indicating that the update has been performed, close the Web browser.

(*) If this tab is checked at this point, the GPS position is registered from now on.

3.1.3.2 Setup of the GPRS link

“Dépannage TELNET” button

- Click on the “Dépannage TELNET” button;

  A DOS command window will open.

A login and a password will be required. The default values are as follows:

Login: rcpsc

Password: RECOPESCA

List of <u>fixed</u> concentrator parameters:

Name of the command	Command in debugging mode **Prefix:** **?= read** **! = write**	Value
IP address of the concentrator	ppp-ip:	192.168.1.10
ISP login	isp-login:	Contact ISP
Password	isp-password:	Contact ISP
DNS address	dns-ip:	194.2.0.20
Name of the SMTP server	smtp-server:	Contact ISP
GPS activation state	gps:	on
Radio power	radio-power:	on

APN number	apn	Internet-entreprise

Settings to modify

Name of the command	Command in debugging mode **Prefix:** **?= read** **! = write**	Value	Comment
Sender name	sender-name:	xxx@yyy.zz	xxx@yyy.zz is the e-mail account associated with the installed SIM card
Password	Smtp-pwd:	Password associated with the e-mail account of the installed SIM card	

- When prompted by the “RCPSC:>” command, enter one of the commands above preceded by “?”to read the value in memory or “!” to write a new value in memory.

Example:

? ppp-ip + Enter: returns the value 192.168.1.10

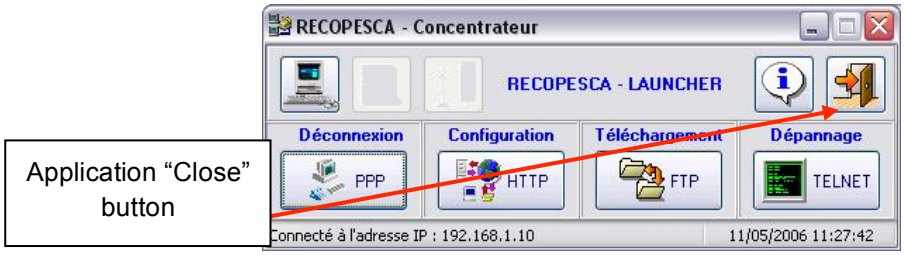
! sender-name:098.recopesca@orange.fr + Enter: indicates that the sender e-mail address to the management station is 098.recopesca@orange.fr

Once all of the verifications and modifications have been performed, exit the” TELNET dépannage” mode:

- At the RCPSC:> prompt, type **!disconnect** + Enter;
- The DOS window will close.

“Déconnexion PPP” button

- Click on the “Déconnexion PPP” button;
- Click on the application “Close” button of the application;



- Disconnect the concentrator from the power supply.

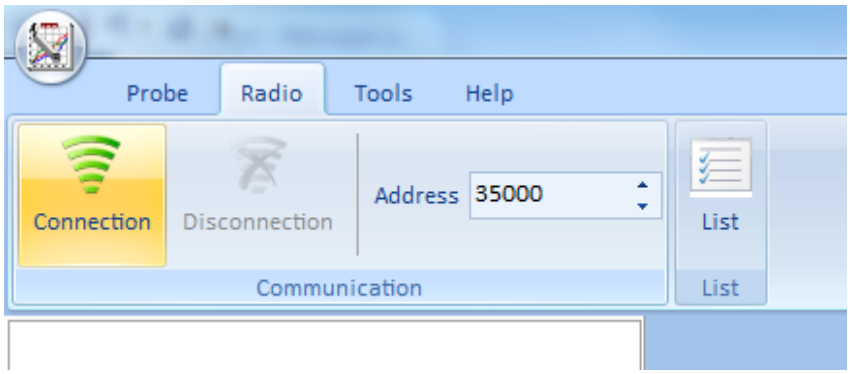
3.2 EAF probes

Required materials

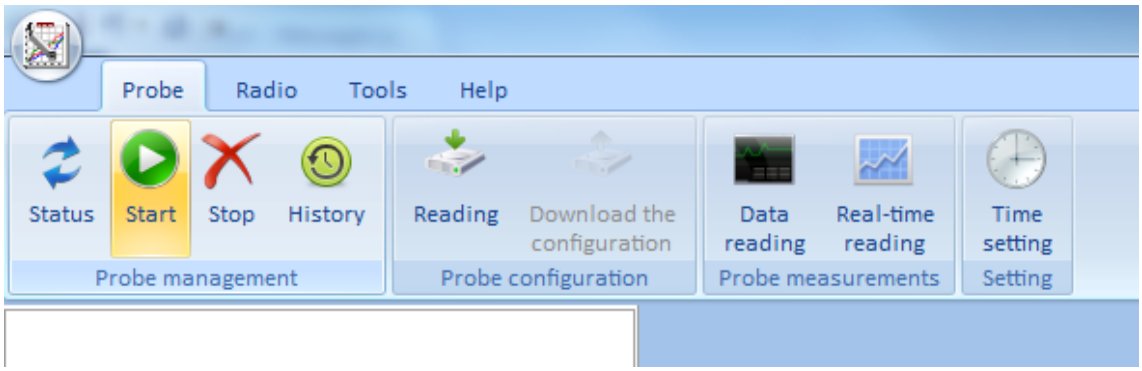
- 1 computer
- 1 Radio Data Pencil
- 1 WINMEMO2 software application
- EAF probes

- Start the WINMEMO2 software application

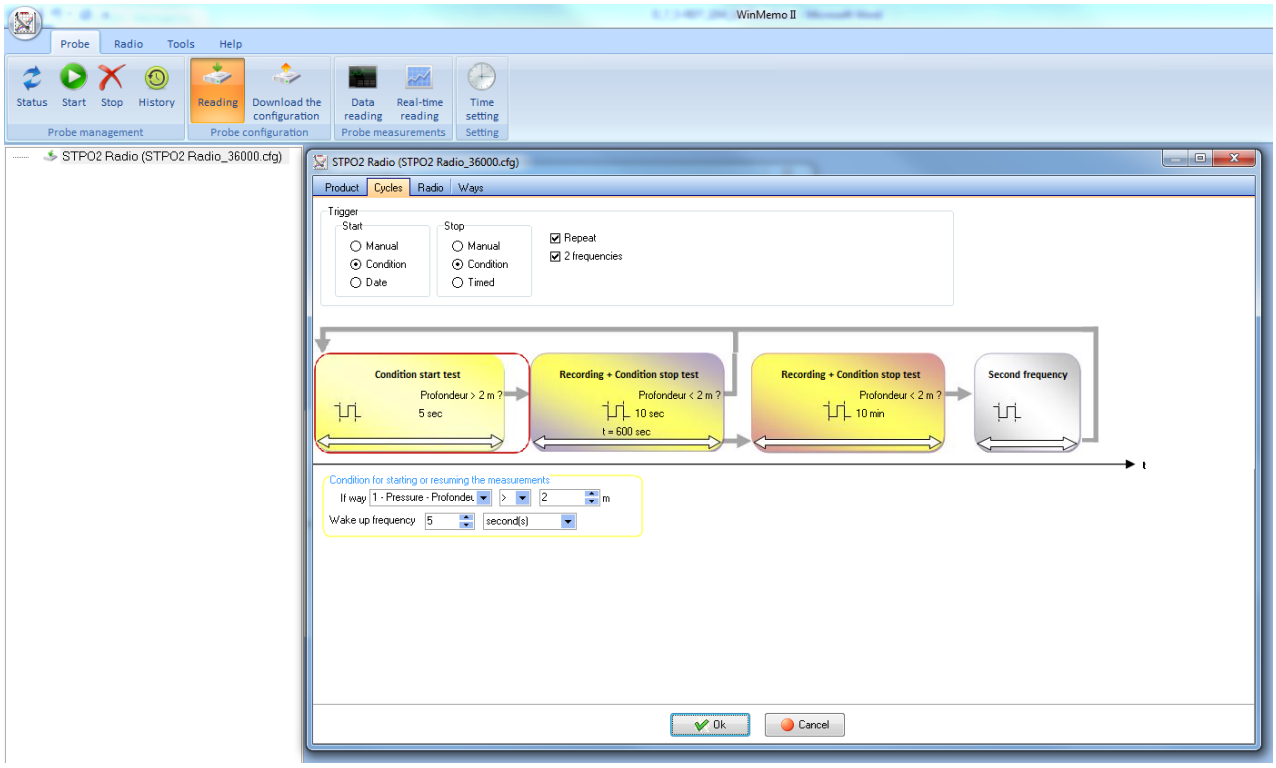
- Connect to a probe (example of a probe radio address:35000);



Click on the “Probe” tab;



- Click on “Reading”;
- Click on the “Cycles” tab;
- Define the parameters of the desired measuring frequency/measurement cycle:

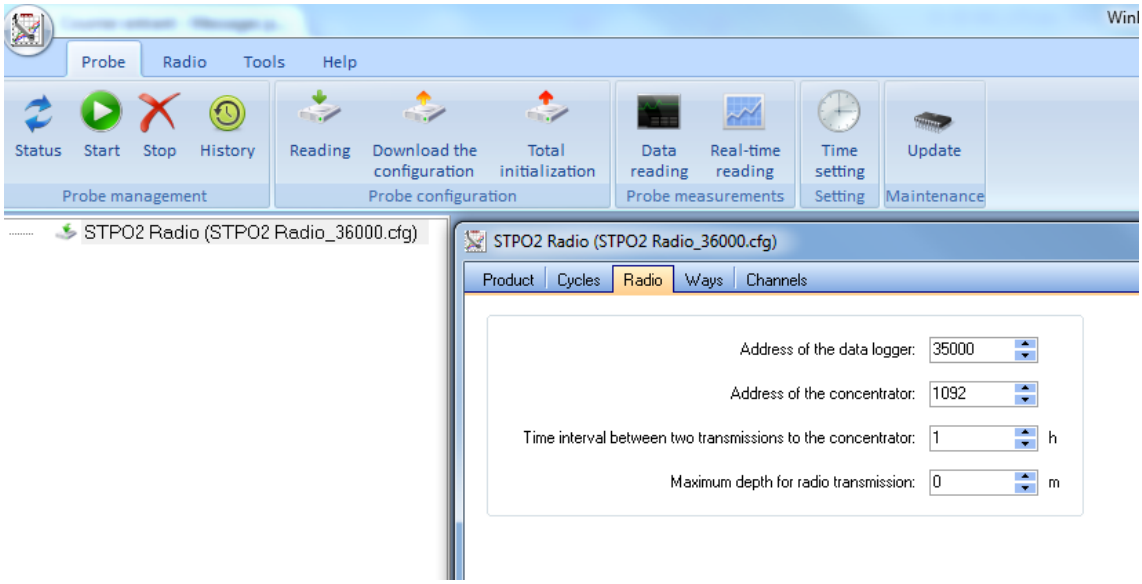


- Click OK.

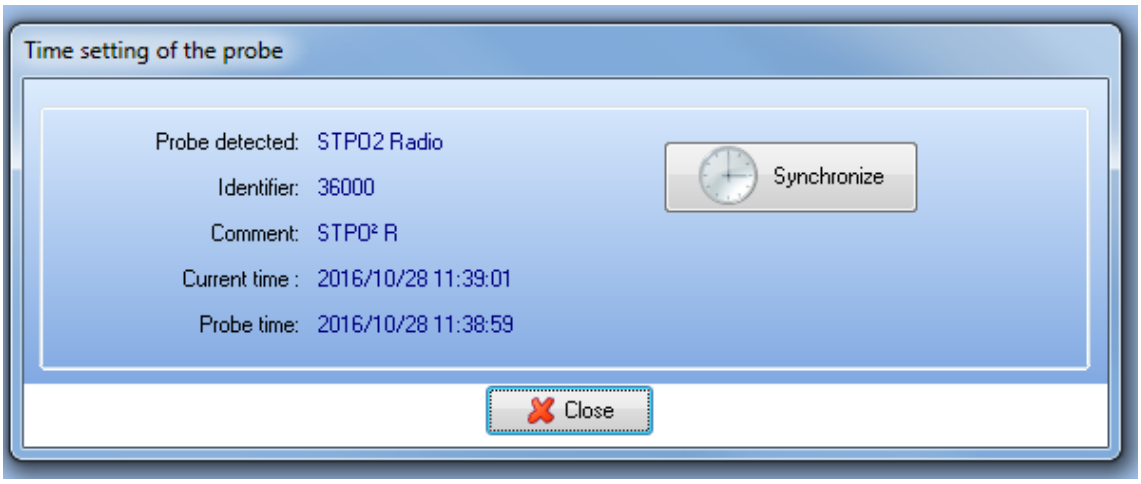
In the example above:

1. Start and stop conditioned on depth (start: >2 meters; stop: < 2 meters);
2. 2 measurement frequencies:
    a. 1st frequency: 1 recording every 10 seconds for 600 seconds;
    b. 2nd frequency: 1 recording every 10 minutes;
3. Cycle begins again when the start condition is met (depth > 2 meters);

- Click on the “Radio” tab;



- Enter the address of the concentrator that the probe transmits to;
- Enter the time interval between two probe data transmissions to the concentrator;
- Click OK;
- Click on ”Time setting”;



- Click on “Synchronize” and then “Close”;
- Click “Start”;
- Click on the “Radio” tab and then the “Disconnection” button.

3.3 Ship-board installation

3.3.1 Description of the actions to carry out the installation of a RECOPESCA GPS-RADIO concentrator on board a fishing vessel

3.3.1.1 Required materials

A RECOPESCA concentrator equipped with :

- SIM card;
- Electric cable H074 (2 x 1.5 mm²) or equivalent;
- Universal controller to verify the polarity of the electric connection;
- A laptop computer equipped with the nke "concentrator.exe" software application and a RECOPESCA network connection;
- An nke Radio Data Pencil.

3.3.1.2 Physical installation of the concentrator

The selected site of installation on the vessel should satisfy the following recommendations:

- Minimise the distance between the concentrator and the measuring probe;
- Avoid direct exposure to heavy sea waves directly on the concentrator;
- Position the concentrator horizontally, open to the sky and open to the horizon on all sides; the cover must face upward to receive GPS satellite data;
- Do not place any metal objects above the device;
- Do not place the concentrator near radio antenna or in a radar swath.

Securing the concentrator:

The housing is secured on a 316L stainless steel plate using 4 CHC M5x40 stainless steel screws A2 with self-locking nuts.

Photo 1: The housing with its support plate. The nuts are visible under the stainless steel plate.

Place a 15 mm thick neoprene shock-absorber (ref: CNA, supplier: Revol Sonier 69516 Vaulx-en-Velin) between the plate and the vessel surface.

Photo 2: concentrator and the shock-absorbing plate.

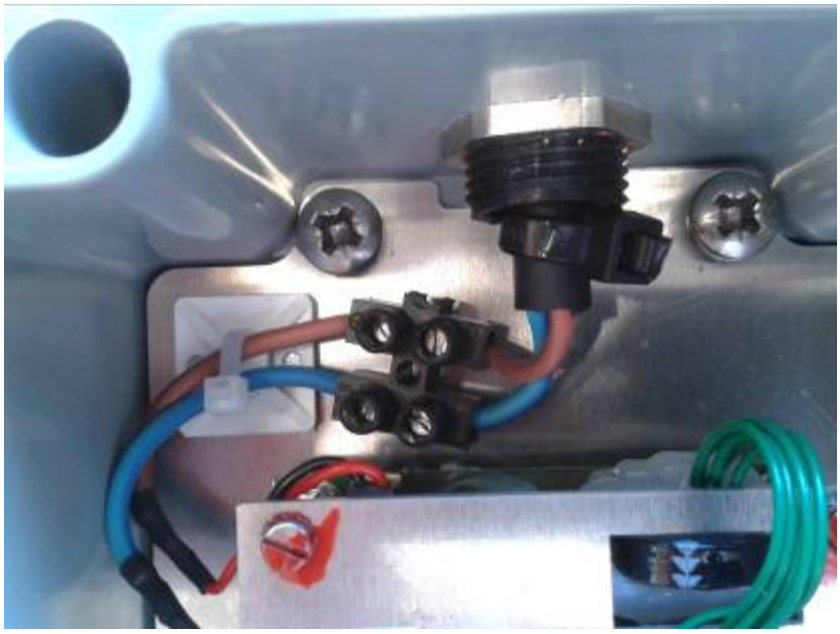
The plate is fastened to the vessel using 4 security screws appropriate for the specific conditions encountered by the vessel.

All of the screw holes made on the vessel to secure the concentrator must be waterproofed with the appropriate sealant.

3.3.1.3 Electrical installation of the concentrator

The concentrator is connected electrically to the ship using a cable threaded through a cable gland.

The electrical connection inside the housing requires an electrical connector.

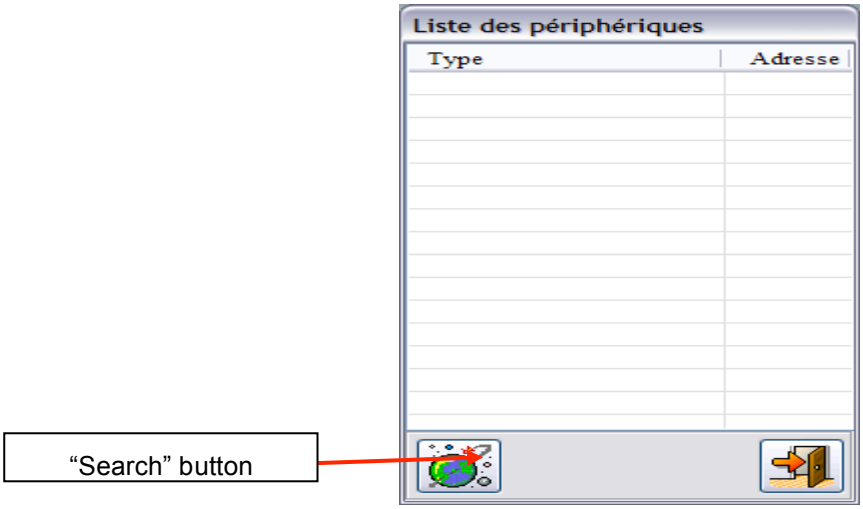


The other end of the cable has to be plugged into the vessel's power supply. The ends of the cables must be fitted with isolating collars.
If possible, ensure that the connector is connected to the vessel's permanent power supply (e.g. back-up batteries). It is absolutely necessary to avoid untimely electric switching that can damage the flash memory of the concentrator.
Electrical polarity must be correct: reversed electrical polarity will destroy the concentrator.
The cable is maintained in place all along its length with mounting clamps appropriate for outdoor use and in sufficient number.
For penetration through walls or partitions, cable glands are used.

3.3.1.4 Turning on the concentrator

The following operations are carried out once the concentrator has been connected to a power supply.

- Start the laptop computer and set the computer's clock to UTC time (GMT+0);
- Launch the concentrator.exe software application;
- Click on the "Scan for devices" button;



- Click on the "Search" button;

A window with the list of detected devices opens:

- Double-click on the name of the concentrator to be set up;

- Click on the "Dépannage TELNET" button;

  A control window will open.

A login and a password will be required. The default values are as follows:

Login: rcpsc

Password: recopesca

- At the RCPSC:> prompt, type **?sntp** + Enter

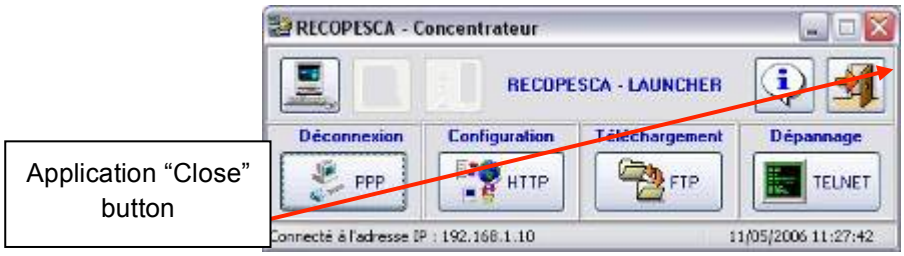
- At the RCPSC:> prompt, type **!disconnect** + Enter;
- The DOS window will close.
- Click on the “Configuration HTTP” button to open the concentrator setup window;

A concentrator setup window opens in your web browser.

- Do not modify the set values;
- Tick the box “Acquisition GPS & envoi e-mail”;
- Click on the “Validation” button.

After receiving a message indicating that the parameter has been updated, close the web browser navigator.

- Click on the “Déconnexion PPP” button;
- Click on the “Close” icon to close the application;



- Close the concentrator.exe software application.

The concentrator is now operational.

3.3.2 Attaching the probe to the fishing vessel

The probe, turned on and calibrated, is placed on the fishing vessel according to the recommendations in the following table:

Type of fishing vessel	Location on fishing gear
Trawler	On the cod end of the trawl net
Creelfishing boat	In a basket trap

Netter	On the ground tackle of the net

4 Calibration

The integration of the EAF STPO2 and STPFluo temperature data into coastal networks is more demanding than the specifications for fisheries studies. The calibration methods were discussed and agreed upon in the FP7 I3 JERICO project (Ref.: JERICO deliverable D4.2).

4.1 Calibration of the EAF STPO$_2$ probe

4.1.1 Depth

Positioning the measuring point is important. Because the location is that of the ship and not of the probe and because time does not correspond to a position, the depth is the only precise reference point for the EAF STPO2 and STPFluo probes.

The general calibration procedure is given in detail in Appendix 1.

The operational depth range is 300 meters for the EAF STPO2 and STPFluo probes.

Most metrology laboratories do not have a manometric balance. Thus, although strictly speaking, a calibrating balance is recommended, this is not always feasible. The following method is the one that will be used the most often. It satisfies our requirements given the level of precision needed.

The method involves placing the probe in a compression chamber. Prior to testing, the probe is placed in a bag filled with demineralised water to avoid any contamination of the optical surfaces by the water in the chamber.

A reference probe calibrated at the IFREMER metrology laboratory is used to assess the accuracy of the pressure sensors in the test probes.

In the range of 0 and 300 dbar, the residual error is checked over pressure levels with the following values:

Measured pressure	Residual error
0	{0.5 – 1 dbar}
50 dbar	
100 dbar	
150 dbar	
200 dbar	
250 dbar	
300 dbar	

4.1.2 Temperature

The integration of the EAF STPO2 and STPFluo temperature data into coastal networks is more demanding than the specifications for fisheries studies. The calibration methods were discussed and agreed upon in the FP7 I3 JERICO project (Ref JERICO deliverable D4.2 - Report on Calibration Best Practices (http://www.jerico-fp7.eu/deliverables/d4-2-report-on-calibration-best-practices)).

The following recommendations are mentioned in JERICO:

- *It is wise to keep an externally calibrated, certified SPRT (Standard Platinum Resistance Thermometer) to use as an independent temperature reference for comparisons if needed; such an SPRT can also serve as a surrogate standard resistance to help check thermometry bridge performance if proper reference resistors are unavailable.*
- *The temperature calibration bath should be allowed to settle at a calibration set-point for a sufficient period of time (an hour or more) before sampling is initiated. The temperature stability and fluctuations of the bath should be continuously monitored during the sampling interval.*
- *The calibrated sensors should be checked at least at a few calibration set-points prior to releasing them for duty. In the case of conductivity, seawater with different salinities could be used in the bath to obtain the necessary calibration set-points although this practice is not commonly followed.*
- *Marine T sensors require regular, often frequent, calibrations because their performances tend to vary over time and can be affected in different ways by specific conditions of usage and/or storage. Sensor calibrations need to be verified at least* ***once a year****.*
- *The results of a calibration may or may not be accredited but they must always be accompanied by the following:*
    - *A declaration of the uncertainty associated with the calibration process;*
    - *Information evidencing traceability to reference material (certified or otherwise): ITS-90 fixed points for temperature and IAPSO Standard Seawater for conductivity.*

The general calibration procedure is detailed in Appendix 2. This procedure is generic; for this specific application, we recommend the following instructions for calibration:

Reference calibration equipment:

- Temperature calibration water bath in which water circulates continuously to maintain uniform temperature. The capacity of the bath must be sufficient to maintain a constant temperature, at least 0.1 m^3. The homogeneity of the bath has to be better than 0.01°C and the stability better than 0.005°C during the calibration period. The water in the bath can be freshwater or natural seawater.
- A sensor to measure the reference temperature. The uncertainty has to be better than 0.01°C. The reference temperature sensor must be calibrated regularly, preferably by a reference laboratory.

Calibration protocol:

- The reference temperature sensor is immersed in the bath;
- The EAF $STPO^2$ probe is placed near the reference temperature sensor;
- During the measurements, the stability of the bath should not exceed ± 0.001°C. The temperature of the room during calibration is set to near 20°C;
- Six temperature set-points are carried out at 0, 5, 10, 20, 25 and 35°C;
- Measurement frequency: 1 measure/10 seconds;
- Measurement time: 15 minutes minimum;

- No self-heating control point;
- No heat leak control point;
- Control point for precision: 20°C.

Calibration results:

- All results should be delivered as a correction to apply to the sensor (either for specific calibration points or over the whole range of temperature covered by the sensor) associated with the uncertainty of the determination of this correction.
- Information demonstrating traceability to SI units ("Système International d'unités") must be provided.

Calibration periodicity:

- At least once a year;
- Preferably just before and just after deployment.

4.1.3 Oxygen

4.1.3.1 Integration of the probes

The STPO2 probes are fitted with an OEM module developed by the PyroScience company, with a temperature sensor and a pressure sensor. The integrated OEM module measures the concentration of oxygen in terms of the % of air saturation and in µmol/L.

The oxygen sensor is calibrated beforehand at two test points and in two solutions of different oxygen concentrations (0% and 100% of air saturation) before integration in the STPO2 housing.

The calibration is performed as follows: the OEM module is connected to the computer via a USB port. The Oxygen Logger application provided by the manufacturer defines the settings of the OEM module (compensation of oxygen measurement with temperature or not) as well as the calibration of the module. Once the module has been calibrated, it is connected to the electronic board of the STPO2 probe. The test points for 100% oxygen at ambient temperature are checked after the STPO2 probe has been installed.

4.1.3.2 Full in-lab calibration

For dissolved oxygen (DO) sensors, the recommended calibration routine relies on comparing the dissolved oxygen measurement of the sensor with Winkler titration lab analyses. To attain the best accuracy possible and to compensate for various effects, a wide range of different concentration levels and different temperature levels must be performed during the calibration.

This protocol, composed of a multi-point calibration followed, if needed, by adjustment, is carried out in compliance with the recommendations of the manufacturer.

The calibration protocol compares the results of DO sensors immersed in water of controlled DO concentrations with reference measurements.

❖ **The reference measurement**

As widely accepted in the scientific community, the reference measurements is Winkler titration**.**

Winkler titration (courtesy of Ifremer).

Originally developed by Winkler in 1888, this method has been adopted by the oceanographic community and is recognized as the most accurate technique to determine dissolved oxygen in seawater.

The term "dissolved oxygen", in common use, refers to the molecule of dioxygen O2 in solution in water.

Since oxygen is a highly reactive compound and, in addition, an atmospheric gas in solution, the method is known to isolate the sample from the air and to fix the dissolved oxygen as rapidly as possible by reaction with a precipitate of hydroxide manganese formed in the sample. By means of a succession of reactions, an iodine solution is obtained that is easily determinable with precision and that is proportional to that of the oxygen initially present.

The iodometric method used is the optimized version by Carpenter (1965a, b) and Carritt & Carpenter (1966). It was adopted very early in this form by the oceanographers of the Baltic Sea (Carlsberg, 1972) and advocated by the Food and Agriculture Organization (FAO) (1975). Used in most laboratories (Levy et al., 1977), it became the international reference for the Woce program (World Ocean Circulation Experts, Culbertson, 1991). Be aware that the international standard ISO 5813 (NF EN 25813, Afnor, 1999c) is not optimal especially for seawater and has therefore been adapted.

Over time, the Winkler protocol has been largely described, improved, and published in several papers (most of them can be found easily on the web):

- Carritt, D.E. 1964. Intercomparison of methods in chemical oceanography. I. Precision and accuracy of the Winkler method. National Academy of Sciences-National Research Council.
- Carpenter, J. H. 1965. The accuracy of the Winkler method for dissolved oxygen. Limnol. Oceanog., 10: 135-140

- Carpenter, J. H. 1965. The Chesapeake Bay Institute technique for the Winkler dissolved oxygen method. Limnol. Oceanog., 10: 141-143
- Culberson, C. H. July 1991. Dissolved Oxygen WHP Operations and Methods
- DOE (1994) Handbook of methods for the analysis of the various parameters of the carbon dioxide system in sea water; version 2. A. G. Dickson & C. Goyets (eds.), ORNL/CDIAC-74
- A. G. Dickson, Determination of dissolved oxygen in sea water by Winkler titration (http://cchdo.ucsd.edu/manuals.html)
- Méthodes d'analyse en milieu marin, Alain Aminot et Roger Kérouel "Hydrologie des écosystèmes marins ; paramètres et analyses" (336 p.)
- Carlberg S.R., 1972. New Baltic manual, with methods for sampling and analysis of physical, chemical and biological parameters, 1972. ICES, Coop Res., 29A
- Levy et al., 1977. The determination of dissolved oxygen in sea water. Bedford Institute of Oceanography. Report Series, BI-R-77-9.

In terms of quality of the measurement, the uncertainty of the Winkler volumetric method can be calculated following the *Guide to the expression of uncertainty in measurement* (JCGM 100:2008). It leads to uncertainties varying between ±2 to ±5 µmol/L and depends on:

- The dissolved oxygen concentration of the sample to be analysed (uncertainties are combined with the volumes of reagents added);
- The operator skills and the performance of the equipment (precision terms).

However, the major component of the uncertainty comes from the uncertainty of the volume delivered by the titrator. This uncertainty component is the volumetric tolerance of the burette, which must comply with regulations.

The units used for dissolves oxygen can be convert using this link: http://envlit.ifremer.fr/var/envlit/storage/documents/dossiers/prelevementhydro/chapitre8.html

❖ The protocol of calibration

The calibration is to be carried out over the range of dissolved oxygen values measured in situ. Depending on the width of this range, several calibration set-points are to be recorded (including the extreme points of the range) at different temperatures corresponding to the range of temperature measured at sea.

Uncertainty estimate

The uncertainty of the calibration is calculated combining the uncertainties components of the Winkler, of the bench and of the sensor as recommended in the Guide to the expression of uncertainty in measurement. When an adjustment is proposed, the uncertainty components related to the way the sensor was adjusted are added to calculate the final adjustment uncertainty.

Comments:

If needed, a specific expertise of the sensor response with regard to pressure could be

also done (Tengberg and Coauthors, 2006). However, this application needs specific pressure facilities.

The adjustment process

In case of adjustment, the protocol to be followed should be the more accurate one recommended by the manufacturer. Indeed, without any published metrological recommendations, the manufacturer practices are the references.

4.1.3.3 Partial calibration control

Because the protocol mentioned above can only be performed by a few laboratories specifically equipped and skilled, a simpler control is usually performed in routine by operators.

It consists in immersing the sensor in a water bath stirred for at least half a day. This bath must be stable in temperature (the use of a temperature regulated bath is recommended). In this case, we assume that the bath is saturated in oxygen and that the percentage of saturation is 100%.

This protocol is also described in the manufacturer user manual.

The value of the percentage of saturation of the bath can then be checked against this assumed value of 100%.

Calibration periodicity: At least just before and just after deployment.

At the end of the first operational deployments on fishing vessels, we will be able to determine the exact metrological performances of the sensors.

4.2 Calibration of the EAF STPFluo probe

4.2.1 Depth

See 4.1.1

4.2.2 Temperature

See 4.1.2

4.2.3 Fluorescence

Among the many calibration methods for fluorimeters studied by JERICO Integrated Infrastructure Initiative for coastal monitoring, the choice is based on instrument type, its compatibility and need for field checks. Use of two or several calibration methods may increase the traceability and the scientific collaborations both in fishery research and coastal operational monitoring. It is too early to be specified and not planned in NeXOS.

The main recommendations are as follows:

- Calibrations must be carried out at constant temperature.

- It is recommended to record fluorescence values with the calibration methods before any coefficients are changed and compared to previous values to assess instrument stability and need for new adjustment.
- If factory calibration is to be overwritten, the user must understand the meaning of calibration procedure. An alternative to changing instrument internal calibration coefficients is to include the new coefficients in data logging or processing software only.
- Calibrations must be made with minimum amount of background light. The effect of light on fluorescence readings should be checked.
- If calibration is done using liquids, glass beakers should be used because plastic materials may cause background fluorescence. Black non-fluorescing material should be used under the glass beakers.
- Calibration using the field housing should be preferred.

The cleanest possible water must be used to check blank values. Care must be taken with that blank values.

As indicated in the title, the “calibration” proposed is in fluorescence. Indeed, currently, there is no possibility to perform recognized, traceable and adequate in-lab calibration of fluorescence sensors in terms of µg/L of chlorophyll *a*.

For this reason, this “calibration” should be considered as a control. The experiment is carried out with fluorophores that are neither traceable, nor recognized and nor generic to all sensors. The control will only lead to the verification of the linearity, the drift and the stability of the sensor between two campaigns.

For this specific application to EAF STPFluo probes, we propose the use of fluoresceine dilutions. Don’t use chlorophyll-a solved in aceton because of the risk of optical damage.

Calibration protocol:

- 5 fluorescein dilutions: 100, 300, 600, 1200 and 2400 µg/L;
- Measurement frequency: 1 measurement/10 seconds;
- Measurement time: 15 minutes minimum;
- Calibrations must be carried out at constant temperature (e.g., temperature regulated lab);
- Calibrations must be made with minimum amount of background light. The effect of light on fluorescence readings should be checked.

Calibration periodicity: we recommend just before and just after deployment.

At the end of the first operational deployments on fishing vessels, we will be able to determine the exact metrological performances of the sensors.

5 Conclusions

At nke, the sensor development phase ended in late May. A new, more appropriate oxygen sensor was selected. Consequently, sensor development required more time than initially scheduled. The qualification tests started late, beginning only in the summer. Ifremer worked intensively in close collaboration with nke to finish this task. We encountered several obstacles. We had some adjustments to make with nke. At the end of the environmental trials, the solar radiation testing damaged both prototype

probes. Analyses and expertise are underway. We believe that the solar radiation tests were too severe for the devices being tested. See Deliverable 7.4 – Acceptance and calibration report.

The sensor prototypes were available again in October 2016. The production of the additional replicate sensors has now been scheduled. They will be available for the sea trials in Italy, Norway and France in early January 2017. Ifremer performed preliminary tests at sea with the collaboration of CNR ISMAR. The tests were done on board the research vessel *G. Dellaporta* on 2-9 November 2016 with the two prototypes.

All the following functionalities of the EAF probes and the whole system with an updated concentrator have been tested:

- Data storage capacity and quality;
- Each function implemented in the firmware that are needed for RECOPESCA (2 cadences, condition-measured activity, resume);
- Typical measurement cycle (usual French RECOPESCA cycles and CNR cycle(s));
- Time stamping and absence of missing data;
- Data transfer to concentrator and quality of the file format & data;
- Data transfer to Data Centre and quality of the data (file format).

The results obtained are in accordance with the technical specifications.

The metrological performances of the sensors were evaluated. The hysteresis and response time were measured during the launching of a CTD probe for pressure, temperature, dissolved oxygen and fluorescence.

The pressure sensor complies with specifications.

The response time in temperature of the two probes did not comply with specifications (<0.5 sec 63%). The temperature sensor must be modified so that its measurements do not vary with the temperature of the probe. A solution similar to that used for the STPS and SP2T probes will be used.

The measurements of oxygen saturation show a large shift that cannot be attributed to an offset. It seems that the discrepancy depends on temperature. The measurements of oxygen concentration (µmol/L) are not consistent with the level of saturation measured. Aberrant values were recorded during measurements of air. Trials back in the laboratory and tests in temperature-regulated baths confirm that the amount of deviation varies with temperature. Studies are underway in collaboration with the oxygen probe manufacturer. The problem appears to be due to poor integration of the

ambient temperature for the calculation of concentration.

The fluorescence sensor recorded a signal comparable to the reference material (fluoresceine). However, the level of fluorescence during this time of the year (autumn) is very low. Other tests are needed during the spring phytoplankton bloom. The STPflu is not a chlorophyll-a sensor, as it's name indicates, it's a Fluorescence sensor. As explain in the joint publication "CALIBRATION PROCEDURE FOR IN SITU MARINE FLUOROMETER, International metrology congress 2003, L. Delauney & Y. Le Guen », Chlorophyll-a sensors doesn't exist! Fluorescence is used as a proxy to achieve estimation of biomass in seawater. In situ, Chl-a fluorescence is not a constant parameter vs biomass quantity, it depends of the site and it depends of the season. Consequently, for formal metrology activity on fluorescence sensors, as demonstrated in the joint publication and sometimes after as reported by the Alliance for Coastal Technology (http://www.act-us.info) in their report on how to manage in situ fluorometer (http://www.actus.info/Download/Workshops/2005/GoMOOS_Fluorometers/index.html?pageNumber=1) it is good practice to use fluorophore for formal fluorometer metrology works and then to use in situ water sampling and Lorenzen chla-a extraction to set up fluorometer to be used on a specific site at a specific moment (season effect). The aim of the work performed here was to demonstrate the metrological performance of the STPflu as a formal metrology qualification, regardless of the site where there will be used. So, fluorophore was the most appropriate procedure to be used.

In December, CNR ISMAR perform further tests at sea.

Highlight clearly significant results:

The first results after the sea trails are promising. The problems in temperature response time and hysteresis have been clearly identified and will be solved.

Reasons for deviations:

Failure to comply with technical specifications for temperature and dissolved oxygen measurements.

Reasons for failing:

No project failure.

Corrective actions:

New design for the mechanical integration of the temperature sensor and for sensor protection. Changes cannot be made yet on the two prototypes. They will be done for the additional replicate sensors. The problems encountered for oxygen measurements are under study. Additional calibrations will be performed on the new version.

6 Reference documents

NEXOS DoW

AFNOR NF X 10-812 Standard — Marine environment – Oceanographic instrumentation – Guide for environmental tests.

EN 1325-1:1997 Standard — Value management, value analysis, functional analysis vocabulary Value analysis and functional analysis. Published*:* January 1997

IEC 60945 Maritime navigation and radiocommunication equipment and systems – General requirements – Methods of testing and required test results

JCGM, 2012. International vocabulary of metrology - Basic and general concepts and associated terms (VIM), in: Metrology, J.C.f.G.i. (Ed.)

JERICO D 4.2 Report on Calibration Best Practices (http://www.jerico-fp7.eu/deliverables/d4-2-report-on-calibration-best-practices).

Measurement of dissolved oxygen concentration. Aminot A., Kérouel R., 2004. Hydrologie des écosystèmes marins. Paramètres et analyses. Éd. Ifremer, 336 p. ISBN2-84433-133-5. Chap VII - p92.

Uchida, H., Kawano, T., Kaneko, I., Fukasawa, M., 2008. In situ calibration of Optode-based oxygen sensors. J. Atmos. Oceanic Tech. 25, 2271-2281.

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Hiroshi, U., Kawano, T., I., K., Fakasawa, M., 2008. In Situ Calibration of Optode-Based Oxygen Sensors. J. Atmos. Oceanic Technol. 25, 2271-2281.

Nelder, J.A., Mead, R., 1965. A Simplex method for function minimization. Computer Journal 7, 308-313.

Appendix 1: PRESSURE CALIBRATION PROCEDURE

Ifremer
Centre de Brest
Service RDT/IC
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Tel.. 33 (0)2.98.22.41.51
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PROCEDURE
OF THE METROLOGY LABORATORY
AT IFREMER, BREST CENTER

Pressure calibration of a pressure sensor against reference standard.

5th edition – April 2010

N° MP004

Only copy

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This document contains 11 pages in all.

1. PURPOSE

The purpose of this document is to describe the procedure to follow to calibrate a pressure sensor with respect to the reference standard, i.e. the Desgranges & Huot 5303 pressure balance.

The information provided here covers the theoretical aspect of calibration.

2. REFERENCES

- **MOpP003:** Use of the Desgranges & Huot type 5303 pressure balance. (document in French)
- **MOpP004**: Preparing and realizing a pressure calibration of a pressure sensing device compared to a Desgranges & Huot type 5303 deadweight tester or pressure balance. (document in French)
- **LAB GTA 11:** Technical Accreditation Guide: pressure metrology. (document in French)
- **Manufacturer's instructions for the device under test.**

3. DEFINING THE CALIBRATION PROGRAM

Before testing a pressure device, the calibration program to be carried out must be defined.

In any case, the calibration program must be adapted as much as possible to the device's later use.

Several steps can be identified:

3.1. DEFINING THE CONDITIONS OF USE FOR DEVICE UNDER TEST

This means defining the specifications of use for the device in order to:

- correctly handle the device,
- adjust the calibration program to make it as close as possible to the device's conditions of use.

At the very least, the following parameters must be identified. This is a non-exhaustive list and of course any other critical parameter must also be defined.

3.1.1. Measurement medium

The client must indicate the measurement medium: air, thermostatic bath (water, oil, any type of fluid, etc.), relying, if needed, on assistance from the calibration laboratory personnel.

It there is no specification from the client, the measurement medium will be air.

3.1.2. Connecting the device

The client must indicate the type of mechanical connection present on the pressure device.

3.1.3. Pressure transfer fluid

The client must indicate the type of transfer fluid accepted by the pressure device.

3.1.4. Device power supply

This paragraph only concerns devices whose power supply (voltage and/or current) can be modified.

Depending on the device, the power supply voltage and/or power supply current must be known, in order to:

- avoid damaging the device,
- realize a calibration which is as close as possible to the conditions of use,
- reduce possible effects related to the device's power supply on the device measurement.

This information must be supplied by the client.

Failing specification from the client, the voltage and/or current of the device's power supply shall be defined by the calibration laboratory staff, taking account of:

- recommendations for use (when manufacturer's or other user manual or corresponding operating procedure has been provided),
- experience the calibrator has acquired for this type of device.

3.1.5. Frequency of acquisition of device measurements

The client must indicate the rate of measurement acquisition.

Failing specification from the client, it shall be defined by the calibration laboratory staff, taking account of:

- recommendations for use (when manufacturer's or other user manual or corresponding operating procedure* has been provided),
- experience the calibrator has acquired for this type of device.

Note:

This paragraph mainly concerns devices whose measurement acquisition is done automatically via a PC software interface.

The very high acquisition frequencies that these devices have lead to issues and additional verifications concerning how meaningful and representative the measurements realized are.

For them, appropriate definition of measurement frequency is important for:

- the similarity of the calibration with the device's conditions of use,
- preventing an error of mismatch between the acquisition rate and the device's response time,
- potentially, verifying the device's ability to detect variations in pressure.

Devices for which acquisition is realized by manual readings of the sensor's indication are generally not affected by the issues outlined above. However, the rules set out at the beginning of the paragraph remain applicable all the same.

3.1.6. Number of measurements per calibration point

The number of measurements made per calibration point will be identical for all calibration points.

The choice of how many measurements are taken is left to the initiative of laboratory staff. However, a few recommendations about choosing the number are given below:

- It is preferable to carry out a series of measurements rather than only one, since the mean of a series will always be closer to the "true value" than a single measurement will be.

- The number of measurements should be set taking account of the frequency of acquisition already established (see paragraph 3.1.5) and so that it is as close to the measurement chosen when the device is in use.

- It is recommended that a minimum of 5 measurements be taken per calibration point. However, a minimum of 3 measurements is possible when the laboratory staff is sufficiently familiar with the type of device under test to anticipate the fact that the measurement will not change and that additional measurements would yield similar results (for instance when the device's resolution does not make it possible to access mesurand variations).

3.2. DEFINING THE CALIBRATION UNCERTAINTY

The required uncertainty of calibration should be specified by the client.

Failing customer specification, the calibration uncertainty will be estimated by the calibration laboratory's staff, taking account of experience acquired on this type of sensor, particularly seeing that the uncertainty sought adequately matches the intrinsic characteristics of the sensor under test.

The calibration uncertainty thus defined will determine which calibration procedure should be used (See paragraph 3.3).

3.3. CHOICE OF CALIBRATION POINTS

The calibration points used depend on the calibration range and uncertainty required by the client: the various calibration procedures in general use are described in more detail below.

Depending on the uncertainty sought for the measurements, three calibration methods can be used (See LAB GTA 11):

- **Method 1**: uncertainty > 0.2% of measurement full scale.

  One cycle of pressure 0 - Pmax at 6 measurement points is realized with a step point at Pmax. This cycle is followed by two rises to 1 pressure at around 50% of Pmax.

- **Method 2:** 0.05 % ≤ uncertainty ≤ 0.2% of measurement full scale

  One cycle of pressure 0 - Pmax at 11 measurement points is realized with a step point at Pmax. This cycle is followed by two rises to 1 pressure at around 20%, 50% and 80% of Pmax.

- **Method 3: uncertainty < 0.05% of measurement full scale**

  Three cycles of pressure 0 - Pmax at 11 measurement points are realized with a step point at Pmax.

However, even if a client's specific requirement does not correspond to these procedures,

calibration can still be realized. Taking it for testing will imperatively require that the client protocol is sufficiently comprehensive to enable correct calculation of uncertainties (particularly that there are points of repeatability and that hysteresis can be calculated if necessary).

3.4. CHOICE OF REFERENCE EQUIPMENT

The laboratory staff are responsible for choosing the reference equipment (i.e. the equipment belonging to the lab) to perform calibration operations.

The choice is based on a combination of several criteria:

- all of the reference instruments must enable compliance with the conditions of use of the device under test,
- all of the reference instruments must make it possible to ensure a level of uncertainty in keeping with the final uncertainty of calibration for the device under test,

  Special case:

  For very high-accuracy devices (particularly those whose characteristics are similar to the reference equipment used in the laboratory) and when there is no in-house possibility at the laboratory to meet the required level of uncertainty for the device under test, the laboratory will be allowed to take on the device for testing there after informing the client of the possibility that it could be calibrated in a more appropriate way by other laboratories.
- all of the reference instruments must enable the points defined in the calibration program to be realized.

  Special case:

  It can be tolerated that a certain number of points of the calibration program are outside of the laboratory's pressure generator's recommended range of operation. The number of these points must not exceed 10% of the total number of points realized, in order to guarantee the correct calculation of the uncertainty components of the calibration.

Appendix 2: **TEMPERATURE CALIBRATION PROCEDURE**

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PROCEDURE

OF THE METROLOGY LABORATORY

AT IFREMER, BREST CENTER

Calibration procedure for a temperature sensing device.

1st edition – January 2016

N° MT017

Only copy

	Written	Validated	Signed	Approved
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Signed				
Date				

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1. PURPOSE AND REALM OF APPLICATION

The purpose of this document is to describe the main steps in realizing a calibration of a temperature sensing device.

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2. REFERENCES

- MG001 - Edition 2 - February 2008: Procedure presenting the general principles of techniques to calculate uncertainty. (document in French)
- MT018 - Edition 1 - February 2008: Procedure for defining corrections and uncertainties which are specific to temperature devices. (document in French)
- BNM monograph n°14 – Simplified techniques to approach ITS-90 International Temperature Scale of 1990 – Editions Chiron. (document in French)
- FD X07-028 - October 2002: Calibration and verification procedure for thermometers: Estimating uncertainties on temperature measurements. (document in French)
- FD X07-029-1 - October 2002: Procedure for calibrating and verifying thermometers - Part 1: calibration and verification procedure for probes and resistance thermometers. (document in French)

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3. TERMS USED

The terms defined below will be marked with an asterisk when used in the document.

Off configuration: configuration in which an instrument should be after being powered off. This configuration involves a number of operations to be performed in order to reach this state.

An example:

> Off configuration* of a thermometer: the thermometer should be clean, powered off, disconnected from its display and stored in a protective sheath.

Immersion (of a sensing device): in this document, the term "immersion" is used in its broad meaning and will also be used when a sensor is being introduced into media other than fluids.

Operating procedure: Set of precisely described operations which are implemented when performing specific measurements according to a given method.

Stabilization (of a measurement or an indication): stabilization of a measurement means the oscillation of the measurement around a mean value which no longer changes.

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4. DEFINING THE CALIBRATION PROGRAM

Before testing a temperature sensing device, the calibration program to be carried out must be defined.

In any case, the calibration program should be as closely adapted as possible to the device's later use.

Several steps can be identified.

4.1. DEFINING THE CONDITIONS OF USE FOR DEVICE UNDER TEST

This means defining the specifications of use for the device in order to:

- correctly handle the device,
- adjust the calibration program to make it as close as possible to the device's conditions of use.

At the very least, the following parameters must be identified. This is a non-exhaustive list and of course any other critical parameter must also be defined.

4.1.1. Measurement medium

The client must indicate the measurement medium: air, water, oil, any type of fluid, etc.

Failing specification from the client, it shall be defined by the calibration laboratory staff, taking account of the experience they have acquired on this type of sensor.

4.1.2. Immersion* of the device

This paragraph only concerns partial immersion* devices.

Correctly defining the immersion depth is important in order to:

- avoid damaging the device,
- realize a calibration which is as close as possible to the conditions of use,
- limit the effects of heat leaks from the device and thus minimize sensor measurement errors (see paragraph 4.3.4).

The immersion* depth should be defined by the client.

Failing specification from the client, the device's immersion* depth will be defined by the calibration laboratory staff, taking account of:

- recommendations for use (when manufacturer's or other user manual or corresponding operating procedure* has been provided),
- experience they have acquired for this type of device.
- as a last resort, and if the elements above are not available, the immersion* depth will be the maximum depth of the measurement element, ensuring that non-watertight parts are not immersed.

Hereafter in this document the depth of immersion* will be designated by the letter *z*.

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4.1.3. Device power supply

This paragraph only concerns devices whose power supply (voltage and/or current) can be modified.

Depending on the device, the power supply voltage and/or power supply current must be known, in order to:

- avoid damaging the device,
- realize a calibration which is as close as possible to the conditions of use,
- limit the effects of the device's self-heating and thus minimize sensor measurement errors (see paragraph 4.3.5).

This information must be supplied by the client.

Failing specification from the client, the voltage and/or current of the device's power supply shall be defined by the calibration laboratory staff, taking account of:

- recommendations for use (when manufacturer's or other user manual or corresponding operating procedure* has been provided),
- experience they have acquired for this type of device.

Hereafter, we the power supply current will be designated by the letter *i*.

4.1.4. Frequency of acquisition of device measurements

The client must indicate the frequency of measurement acquisition.

Failing specification from the client, it shall be defined by the calibration laboratory staff, taking account of:

- recommendations for use (when manufacturer's or other user manual or corresponding operating procedure* has been provided),
- experience they have acquired for this type of device.

Note.

This paragraph mainly concerns devices whose measurement acquisition is done automatically via a PC software interface.

The very high acquisition frequencies that these devices have, lead to issues and additional verifications concerning how meaningful and representative the measurements realized are.

For them, appropriate definition of measurement frequency is important for:

- the similarity of the calibration with the device's conditions of use,
- preventing an error of mismatch between the acquisition frequency and the device's response time,
- potentially, verifying the device's ability to detect variations in temperature.

Devices for which acquisition is performed by manual readings of the sensor's indication are generally not affected by the issues outlined above. However, the rules set out at the beginning of the paragraph remain applicable all the same.

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4.1.5. Number of measurements per calibration point

The number of measurements made per calibration point will be identical for all calibration points.

The choice of how many measurements are taken is left to the initiative of laboratory staff. However, a few recommendations about choosing the number are given below:

- It is preferable to carry out a series of measurements rather than only one, since the mean of a series will always be closer to the "true value" than a single measurement will be.
- The number of measurements should be set by taking account of the rate of acquisition already established (see paragraph 4.1.4) and of the fact that it is preferable to have a set of measurements covering at least one cycle of adjustment of the temperature calibrator. Indeed, if the series of measurements is taken on rising (or lowering) temperature, since the responses of the device under test and of the reference instrument do not necessarily have the same response times, a measurement lag between the two instruments may be seen. This difference will be much less if a full temperature adjustment cycle is taken into consideration.
- It is recommended that a minimum of 5 measurements be taken per calibration point. However, a minimum of 3 measurements is possible when the laboratory staff is sufficiently familiar with the type of device under test to anticipate the fact that the measurement will not change and that additional measurements would yield similar results (e.g. when the device's resolution does not make it possible to access mesurand variations).

4.2. DEFINING THE CALIBRATION UNCERTAINTY

The required uncertainty of calibration should be specified by the client.

Failing customer specification, the calibration uncertainty will be estimated by the calibration laboratory's staff, taking account of experience acquired on this type of sensor, particularly seeing that the uncertainty sought adequately matches the intrinsic characteristics of the sensor under test.

The calibration uncertainty thus defined will determine which calibration procedure should be used.

4.3. CHOICE OF CALIBRATION POINTS

The calibration points to realize are chosen by combining the points identified in the following paragraphs.

4.3.1. Points of use for device under test

These are the temperature points covering the sensor's range of use.

These points should be defined by the client depending on how the device under test is to be used.

If the client does not provide requirements, the calibration points will be defined by the laboratory staff in compliance with the following rules:

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In the case when the device is used for spot temperature measurements:

- as many calibration points as points of use.

In the case when device is used over a range of temperature:

- 2 points corresponding to the upper and lower limits of the realm of use,
- at least 1 additional point to check the linear behaviour of the device under test (if this is the case); this point is generally taken at the centre of the sensor's range of use (or at regularly spaced points in the range if more than one point is used).

  Note.

  Adapt the number of additional points to the type of sensing device response depending on the temperature (2 if response is 2nd order polynomial, …, n if response is polynomial of an order of n).

- Recommendations concerning the spacing of points:
  - On temperature ranges very close to ambient conditions (-10°C to +40°C), the calibration points will be spaced no more than 5°C apart.
  - On temperature ranges close to ambient conditions (-20°C to +100°C), the calibration points will be spaced no more than 10°C apart.
  - Beyond that, the spacing is left to the initiative of laboratory staff.

4.3.2. The reference point

In order to verify that no change (damage or structural alteration) has occurred on the calibrated device, it is recommended that within the calibration procedure the same point be realized several times. This is called the reference point.

An example:

- for calibrating thermometers at very low or very high temperatures, going to these temperatures can entail structural modifications of the device and consequently of its response,
- for calibrating thermometers which are sensitive to shocks or other elements (humidity, etc.),
- for calibrating liquid-in-glass thermometers in order to ensure that there is no separation of the liquid column, liquid gone back into the bulb[1] or changes in the properties of the glass capillary.

Thus, the aim of realizing this point is to identify defective sensing devices, i.e. those displaying suspicious variations in reference point indications compared with the stability of the temperature calibrator, the resolution of the calibrated sensing device and the expected calibration uncertainty for this device.

1 i.e. the liquid reservoir

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4.3.2.1. Choosing the point

The choice of reference point is determined by the laboratory staff, following the recommendations below.

Generally and unless there is an indication to the contrary related to the equipment's conditions of use, this point is zero degree Celsius.

It is strongly recommended to utilize a melting ice point or a triple point of water, both more reproducible and more thermally stable than a zero degree Celsius point realized using temperature controlled equipment, particularly in the case of temperature sensing devices whose resolution is similar to the stability of the controlled instrumentation.

Note:

For calibrating liquid-in-glass thermometers, the choice of the ice melting point is commonly used.

4.3.2.2. Choosing the number of repetitions

The reference point is realized at the start and end of calibration and each time that a risk is identified during calibration (as judged by the laboratory staff).

For example:

For calibration to realize these points:

-40°C, -80°C, 0°C, +50°C and +100°C,

the calibration program will be:

0°C, -40°C, -80°C, 0°C, +50°C, +100°C and 0°C.

Note:

Special case of liquid-in-glass thermometers with graduation exceeding 300°C:

The first reference point will immediately be followed by the highest temperature point, then the reference point will be reiterated. Defective thermometers can quickly be excluded thanks to this check.

4.3.3. The control point for precision

The precision of the device is estimated by the scatter of measurements performed during repetition of a point in the calibration program. Checks can also be made at several points of the program, but this is generally not necessary.

The scatter is estimated by statistical data analysis. That is why it is necessary to ensure that some conditions required for statistical application have been fulfilled: one of them is the independence of measurements, i.e. the absence of any link between measurements (or the absence data redundancy between measurements).

To support this hypothesis, theoretically it would be necessary that the measurement point repetition take place after the variation of the mesurand and return to the control point following the same sequence.

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However, in practice it is tolerated:

- either to obtain measurements that are separated by withdrawing then reinserting the tested device during the same measurement point, since this operation is the equivalent of mesurand variation,
- or, in the case of measurements taken over very long time periods, to avoid having to multiply these times, to consider that the temperature calibrator has generated a modification in the mesurand due to its temperature adjustment cycle. That also requires that the frequency of acquisition defined in paragraph 4.1.4 be consistent to ensure that data are independent.

Note 1:

Of these two possibilities, we advise using the former, since there are fewer handling parameters to be defined.

Note 2:

For calibration points a few degrees apart (as recommended in this procedure), there is a strong assumption that the fact of repeating the measurement point (after variation of the mesurand) following a **different sequence** would have very little influence on the sensing device response. Therefore, this practice will be tolerated, especially if it optimizes the calibration time.

4.3.3.1. Choosing the point

The precision control point is one chosen by laboratory staff amongst the calibration program points, following the recommendations below.

- Precision should be tested at the most extreme temperature point.

  Note:

  However care should be taken in applying this rule, since the most extreme temperature points may also correspond to the temperature points for which the temperature calibrator's stability is worst. In that case, the precision measured might only be the stability of the measurement medium.

  That is why, in this specific case, we rather advise to choose the temperature point at which the temperature calibrator is most stable.

- It can be judicious to take the reference point as the precision control point, since it is repeated several times in the calibration program.

4.3.3.2. Choosing the number of repetitions

The number of repetitions of the precision control point is chosen by the laboratory staff, following the recommendations below.

Theoretically, there should be at least 30 repetitions of the measurement point in order to perform classic statistical processing. However, other processing can be performed when fewer data are available.

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In practice, the number of repetitions of the measurement point will depend on:

- the repetition method chosen: repeating the measurement point after changing the mesurand, reinserting the sensor within the same point or with a long acquisition time within the same point.
- the type of measurement acquisition: interfaced (automated) or manual.

General rules:

The choice of the number of repetitions will depend on what the laboratory staff know about the sensing device under test. A few recommendations are set out below for the minimum number of repetitions to perform. However, this minimum number can be raised (without limitation) whenever the lab staff considers it necessary (e.g. to answer any questions about the way the sensor is functioning).

In the case of (automated or manual) acquisitions over long time periods, the number of repetitions is the number of measurements realized within a calibration point.

In the other cases, the number of repetitions is different from the number of measurements realized within a calibration point and the number of measurements performed during the repetitions may differ from the number of measurements realized within a calibration point.

For long acquisition times within the same point:

For automated acquisitions, the number of measurements can be high (more than 30) or even very high (more than 100) since the measurement conditions make it possible.

However, be careful that the total measurement time actually includes the variation of the mesurand (by covering a temperature adjustment cycle) and that the frequency of acquisition also makes it possible to assume that data are independent: insofar as possible, give preference to acquisition rates lower than 1 measurement every 10 seconds.

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For other cases:

The number of repetitions and of repeat measurements will be set jointly in order to optimize the handling time. They will also be set to be consistent with the number of measurements taken at the calibration points themselves.

Number of repetitions:

Seeing the handling times required, significantly reducing the number of repetitions can be tolerated. This number should fall between 5 and 15 repetitions. A minimum of 3 measurements is possible, but only when the laboratory staff is sufficiently familiar with the type of device under test to anticipate the fact that additional measurements would yield similar results (e.g. when the device's resolution is much higher than the variations of the mesurand).

Number of measurements per repetition:

The same number of measurements will be taken at each repetition. The greater the number of repetitions, the smaller the number of measurements per repetition can be, to avoid overloading the trials.

The choice of the distribution of the number of repetitions / number of measurements per repetition is left up to the laboratory staff.

Here are two possibilities for small numbers of measurement per calibration point:

1/ Realizing as many measurements per repetition as measurements per calibration point This means the series of measurements to be analyzed can be compared.

2/ Another pragmatic approach can be to perform one measurement per repetition and as many repetitions as there are measurements per calibration point. Thus, computing the measurement scatter within a calibration point and computing the measurement scatter through repetitions will be comparable, because based on the same number of measurements.

Note 1:

Data scatter computations are not described here because that are not part of the subject for this procedure. Other documents deal with this processing (see documents mentioned in paragraph 2 and especially procedure MG001).

Note 2:

The variance of measurements is a statistical estimator for measurement scatter. This calculation involves a division by the number of measures realized. This highly interesting rule could encourage taking a exaggeratedly large number of measurements with the sole goal of lowering the scatter value. However, we strongly recommend against this practice, which should be used with care with regard to the concept of independent data.

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4.3.4. Heat leak control point

In the context of monitoring heat exchanges between the sensing device and the surrounding environment, it is necessary to evaluate heat leaks propagated by the sensor's sheath.

This is assessed by checking the measurement variations related to variation of immersion* of the device under test.

4.3.4.1. Devices concerned

Assessments of heat leaks only concern partial immersion* sensors and not liquid-in-glass thermometers with a set immersion (immersion to read degree[2], specified immersion depth[3], or total immersion).

4.3.4.2. Choosing the point

The heat leak control point is one chosen by laboratory staff amongst the calibration program points, following the recommendations below.

Generally, the point whose temperature is furthest from that of the ambient environment is chosen.

This point does not add an additional point to the program, but does add tests on one of the already existing points.

4.3.5. Self-heating control point

In the context of monitoring heat exchanges between the sensing device and the measurement medium, it is necessary to evaluate the heat exchanges linked to the sensing devices own heating.

Assessment of self-heating is done by measuring variations in temperature related to the variation in the intensity of the power supply of the device under test.

A practical choice of currents consists in realizing temperature measurements at current i then at current $i \quad \sqrt{2}$ (see procedure MT018 for more information).

4.3.5.1. Devices concerned

This point of control will only concern sensing devices whose sensor element is made up of a resistance that is powered by current in order to measure it[4].

Furthermore, depending on the evaluation technique used, self-heating will only be checked for sensing devices whose power supply is variable or can be modified (voltage source outside of the measurement chain).

4.3.5.2. Choosing the point

The self-heating control point is one chosen by laboratory staff out of all of the calibration program points.

This point does not add an additional point to the program, but does add tests on one of the already existing points.

2Also called total
3Also called partial
4A resistance measurement can only be taken by sending current through the resistance, which produces dissipation of energy and therefore local heating.

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4.4. ORDER OF CALIBRATION POINTS

Before beginning the trials, it is advised that the calibration points be put in an order which enables taking advantage of the fastest temperature kinetics between heating or cooling (generally this will be heating).

So the order of calibration points will be:

- first point: reference point,
- second point: lowest or highest temperature point,
- intermediate points: defined in order to optimize the adjustment kinetics (no requirements as to positioning the control points for precision, heat leaks or self-heating).
- last point: reference point.

4.5. CHOICE OF REFERENCE EQUIPMENT

The laboratory staff are responsible for choosing the reference equipment (i.e. the equipment belonging to the lab) to realize calibration operations.

The choice is based on a combination of several criteria:

- all of the reference instruments must enable compliance with the conditions of use of the device under test,

  Note:

  Insofar as possible, the temperature calibrator should be chosen to entirely hold the temperature sensing device under test in its working range.

- all of the reference instruments must make it possible to ensure a level of uncertainty in keeping with the final uncertainty of calibration for the device under test,

  Special case:

  For the highest-accuracy measuring devices (particularly those whose characteristics are similar to the reference instruments used in the laboratory) and when there is no possibility either in-house at the laboratory or in outside metrology laboratories to meet the required level of uncertainty for the device under test, the laboratory will be allowed to take on the device for testing there provided that the client has been informed.

- All of the reference instruments must enable the points defined in the calibration program to be realized.

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- All of the reference instruments must enable the points defined in the calibration program to be realized.

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5. PREPARING THE CALIBRATION

If possible, the following actions should be carried out within a certain time before the actual calibration starts. We will call this the "time before measurement" (see time called A in the kinematics shown in Figure 1).

Note:

All of the times mentioned in this document can be estimated either as a specific time value (e.g. 4 hours), or by a time interval (e.g. between 4 and 6 hours).

We strongly recommend this practice, since:

- it will enable the laboratory to keep a margin of tolerance for deadlines,
- in the case of temperature calibrators, a single time will cover the entire temperature range that can be realized.

- Start up the temperature calibrator at the first calibration temperature desired (see manufacturer's or other user manual or corresponding operating procedure*).
- Unless otherwise indicated, insert the device under test, complying with the immersion* depth defined.

  Indications to the contrary correspond to risks of damage to the device.

  Some examples:

  - risk of corrosion,
  - risk of losing water or air tightness of device,
  - problem of humidity,
  - risk of physical damage to the device.

  To achieve the most consistent conditions, it is recommended that the probe or sensor of the device under test be positioned at the point used as reference when the temperature calibrator's homogeneity was determined (generally this is at the centre of the temperature calibrator).

  Note 1:

  For liquid-in-glass thermometers, the verticality of the thermometer is extremely important for taking correct measurements.

  Note 2:

  For partial immersion liquid-in-glass thermometers (immersion to the degree read or to a specified depth), a temperature sensing device should be placed near the emergent stem[5].

- Unless otherwise indicated, power up the sensing device under test in compliance with the power supply conditions set out.

5 Emerging from the bath

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Indications to the contrary involve either risks of damage to devices or other recommendations for use.

Some examples:

- damage related to the risk of power cuts,
- damage due to overrunning the measurement range (galvanometer optical scanners),
- for battery-powered sensing devices, to avoid discharging the battery.

➢ Unless otherwise indicated, insert the reference sensing instrument, complying with the recommendations for its immersion* (see manufacturer's or other user manual or corresponding operating procedure*).

In order to achieve the best conditions of homogeneity, it is recommended that the reference device be placed close to the device under test.

Indications to the contrary are the same as those for the device under test.

➢ Unless otherwise indicated, power up the reference sensing instrument, complying with the recommendations for its power supply (see manufacturer's or other user manual or corresponding operating procedure*).

Indications to the contrary are the same as those for the device under test.

In analyzing the preparation steps, it is clear that the "time before measurement" should be determined based on the most disadvantageous of the following characteristics:

➢ temperature calibrator: time to reach a temperature step,

➢ reference instrument: heating time and thermal inertia,

➢ device under test: heating time and thermal inertia,

Since the "time before measurement" depends on the equipment in place, it will be:

➢ either taken from the specific operating procedures* for the realization of the calibration underway when available,

➢ or determined by the laboratory staff during the calibration based on the characteristics corresponding to each piece of equipment: the characteristics of the laboratory's own equipment will be available in the files for each piece of equipment and the characteristics of the device under test will come either from the documents provided or from the experience acquired by the laboratory staff.

If it is impossible to comply with the time, the preparation of the calibration could be directly followed by its realization, providing that the heating time and thermal inertia of the sensing devices are respected.

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Some examples:

- damage related to the risk of power cuts,
- damage due to overrunning the measurement range (galvanometer optical scanners),
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➢ temperature calibrator: time to reach a temperature step,

➢ reference instrument: heating time and thermal inertia,

➢ device under test: heating time and thermal inertia,

Since the "time before measurement" depends on the equipment in place, it will be:

➢ either taken from the specific operating procedures* for the realization of the calibration underway when available,

➢ or determined by the laboratory staff during the calibration based on the characteristics corresponding to each piece of equipment: the characteristics of the laboratory's own equipment will be available in the files for each piece of equipment and the characteristics of the device under test will come either from the documents provided or from the experience acquired by the laboratory staff.

If it is impossible to comply with the time, the preparation of the calibration could be directly followed by its realization, providing that the heating time and thermal inertia of the sensing devices are respected.

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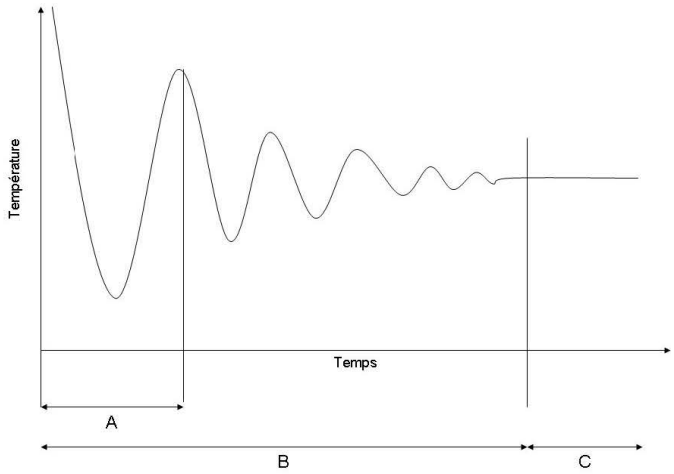

Figure1: Kinetics of adjusting a temperature step in a temperature controlled bath.
Showing the various times to comply with during calibration:
A = time before measurement / B = adjustment time / C = stabilization time.

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6. REALIZING THE CALIBRATION

The steps below will follow the preparation of the calibration:

- If it has not already been done (due to counter-indications related to devices), leaving the necessary heating time and thermal inertia for the sensing devices before starting the measurements:
  - Place the device under test into the temperature calibrator, in compliance with the recommendations for its immersion*.

    To achieve the most consistent conditions, it is recommended that the probe or sensor of the device under test be positioned at the point used as reference when the temperature calibrator's homogeneity was determined (generally this is at the centre of the temperature calibrator).
  - Power up the device under test in compliance with its power supply recommendations.
  - Insert the reference sensing instrument, complying with the recommendations for its immersion* (see manufacturer's or other user manual or corresponding operating procedure*).

    In order to achieve the best conditions of homogeneity, it is recommended that the reference device be placed close to the device under test.
  - Power up the reference sensing instrument, complying with the recommendations for its use (see manufacturer's or other user manual or corresponding operating procedure*).
  - Begin the measurements of the reference instrument.
- Check that the temperature calibrator's adjustment phase has been reached: to do so, verify that reference temperature measurements are stabilized*.

  If there is no sign of stabilization* of temperature measurements after a certain time (see time called B on the kinetics displayed in Figure 1), stop the trials and see why there is no adjustment. We will call this the "adjustment time", counting from when the temperature calibrator has been switched on. Find the "adjustment time" value in the specific file of the calibrator for the temperature chosen.
- Using a reference temperature reading, check whether the "criterion for measurement triggering" has been met. This criterion is a temperature threshold expressed in Celsius degrees below which the temperature calibrator's stability should fall.

  Checking this criterion can ensure that all of the equipment is within a controlled configuration, because it is known and characterized. This criterion guarantees the reliability and repeatability of trials.

  The criterion value is provided in the specific file of the temperature calibrator.

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6.1. MEASUREMENT TRIGGERING CRITERION IS NOT MET

If the criterion for triggering measurements has not been satisfied, continue the test up to a maximum time which we will call the "stabilization time" (see time called C on the kinetics shown in Figure 1). The time is counted from the start of measurement stabilization. Find the " stabilization time" value in the specific file of the calibrator for the temperature chosen.

The test can be pursued either continuously or in stages during this lapse of time (depending on the recommendations for use of the reference instrument).

When the "stabilization time" is exceeded without the calibration triggering criterion being validated, stop the trials and look for the reason by the criterion cannot be met.

6.2. MEASUREMENT TRIGGERING CRITERION IS MET

When the criterion for triggering measurements has been satisfied, taking measurements for the calibration can begin:

- When possible, perform measurements on the reference sensor and on the sensing device under test simultaneously.

  If not, frame the series of measures realized by the device under test by two series of measurements on the reference instrument (one before and one after).

  Note 1:

  When the calibration results are processed the value selected for the reference measurement will be the mean of the two series.

  Note 2:

  For liquid-in-glass thermometers, before any measurement tap the thermometer (e.g. with a pencil) in order to relax the internal constraints which can lead to errors of capillarity.

- Read and note in the documents provided to this end, all indications, whether principal or secondary data, to ensure the traceability of the data or the calculations made.
- Once all measurements have been completed (for this set-point temperature):
  - Leave the device under test in place and powered up (unless otherwise indicated in the text above).
  - Leave the reference instrument in place and powered up (unless otherwise indicated exactly as for the device in test or if the reference sensing instrument is needed for another trial).
  - Go on to a new set-point, wait for the "time before measurement" and repeat the steps given in chapter 6.

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