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Report on the demonstration of silicate sensor on ARGO float in the Mediterranean Sea

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R	Report	x
DEC	Websites, patents, filing, etc.	
DEM	Demonstrator	
O	Other	

Dissemination level		
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CO	Confidential, only for members of the consortium (including the Commission Services)	

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NAUTILOS - New Approach to Underwater Technologies for Innovative, Low-cost Ocean observation is an H2020 project funded under the Future of Seas and Oceans Flagship Initiative, coordinated by the National Research Council of Italy (CNR, Consiglio Nazionale delle Ricerche). It brings together a group of 21 entities from 11 European countries with multidisciplinary expertise ranging from ocean instrumentation development and integration, ocean sensing and sampling instrumentation, data processing, modelling and control, operational oceanography and biology and ecosystems and biogeochemistry such, water and climate change science, technological marine applications and research infrastructures.

NAUTILOS will fill-in marine observation and modelling gaps for chemical, biological and deep ocean physics variables through the development of a new generation of cost-effective sensors and samplers, the integration of the aforementioned technologies within observing platforms and their deployment in large-scale demonstrations in European seas. The fundamental aim of the project will be to complement and expand current European observation tools and services, to obtain a collection of data at a much higher spatial resolution, temporal regularity and length than currently available at the European scale, and to further enable and democratise the monitoring of the marine environment to both traditional and non-traditional data users.

NAUTILOS is one of two projects included in the EU´s efforts to support the European Strategy for Plastics in a Circular Economy by supporting the demonstration of new and innovative technologies to measure the Essential Ocean Variables (EOV).

More information on the project can be found at: http://nautilos-h2020.eu/.

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Executive Summary

The main objective of Task 7.4 (Demonstration on ARGO platform) was to demonstrate the use of the NAUTILOS silicate sensor, developed in task 4.2, on a new generation of ARGO float, the CTS5. This new generation of float equipped with a silicate sensor will ultimately help to increase our understanding of major biogeochemical cycles and their impact on climate. Considering the very high spatial and temporal variability found in open ocean, in situ autonomous sensors implemented with underwater vehicles are the only sustainable solution.

The present document is the final report of Task 7.4 and reports results of the work carried out by partners from M36 up to M57, illustrates further developments, issues encountered, solutions adopted and data exchange with the NAUTILOS data stream services.
Therefore, it is organised in four main sections:
Chapter I: Introduction, provides a brief description of the idea behind the planned activities, some references to the state of the art and a list of the carried-out activities;
Chapter II: ARGO float Demonstration Report, contains details on demonstrations relating to the use of ARGO profiling float, carried out by NKE in the Mediterranean Sea., with the support of CNRS-LGC;
Chapter III: Ethical Considerations, reports considerations on some ethical aspects relevant to the activities described in this deliverable;
Chapter IV: Summary, closes the report, highlighting the main achievements obtained in T7.4.

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No tables.

List of Acronyms and Abbreviations

Abbreviation	Definition
Argo	International programme for researching the ocean
CNRS	French National Centre for Scientific Research
LGC	Chemical Engineering Laboratory
DT-INSU	Technical Division of the National Institute of Sciences of Universe
IFREMER	French Research Institute for Exploitation of the Sea
SWV	Square-Wave Voltammetry
TRL	Technology Readiness Levels
WP	Work Package
CSV	Comma-Separated Values
CTD	Conductivity, Temperature and Depth
GPS	Global Positioning System
NRT	Near Real Time

I. Introduction

NAUTILOS project aims to fill existing gaps in marine observation and modelling through the development of new technologies and their development on different observational platforms. One of the specific objectives of the project is to promote innovative and cost-effective methods in a wide range of critical environmental contexts and European Union (EU) policy applications as for example the Marine Strategy Framework Directive. In this context, NAUTILOS allows the development and validation of technological innovations needed to support the growing demand for wider, cost-effective global ocean and coastal observing systems that establish environmental baseline conditions, detect and monitor environmental changes and underpin the provision of primary environmental services.

Based on the experience of CNRS-LGC (Barus et al, 2021) and NKE Instrumentation (SenseOCEAN, OceanSensor projects), a new generation of electrochemical sensors has been designed, implemented and tested within NAUTILOS to collect measurement of Essential Ocean Variables (EOVs) such as silicate by exploiting the capacity of ARGO profiling floats for ocean observation.

The aim of deliverable D7.5 is to evaluate the performance of a silicate sensor integrated with a profiling float in a real environment. The demonstration took place in the Mediterranean Sea, an environment with marine currents well-studied by scientists. An initial demonstration was held in the summer of 2024 in the Mediterranean Sea, but a problem with the GPS antenna meant that the mission had to be cancelled just before the launch. The risk of losing the float was too high. Prior to this, a series of tests were carried out in Ifremer's seawater tank, enabling all the silicate sensor's functions to be tested. A second demonstration took place in the Mediterranean Sea close to Villefranche-Sur-Mer in April 2025.

II. Argo Float Demonstration Report

1. Background

A first-generation silicate sensor mounted on a profiler was developed as part of the FP7-SenseOCEAN and MarTERA OceanSensor projects. The technology of the time did not allow rapid measurements to be taken to produce acceptable profiles with a maximum number of points in the water column. It took 45 minutes to acquire a measurement. We therefore worked on the mechanical architecture of the sensor to reduce this acquisition time. This work was carried out as part of task 4.2 of the NAUTILOS project. A silicate sensor requires an initial phase of oxidation of the molybdenum to form a complex with the silicates in the medium that can be detected with electrochemical method known as square-wave voltammetry (SWV).

2. Demonstration Plan

When deploying a profiler at sea, there are several validation stages to be considered. After laboratory calibration (ST6.2), integration and field validation (ST5.4), the silicate sensor, developed in task 4.2 was mechanically and electronically integrated on an ARGO profiling float. The first demonstration was planned in a seawater pool at Ifremer to check the functioning of the silicate sensor during the descent and ascent of the float, verify the communication between sensor and float, and monitor the behavior of the float buoyancy. The second demonstration was planned in the Mediterranean Sea by NKE and CNRS-LGC. This site was chosen because of its known marine currents and high depths, which allow us to control the float's trajectory in the Mediterranean Sea in advance and not to lose the float during the mission. This demonstration site was selected to test the silicate sensor up to 1000 m of water depth. The float's mission was configured in advance to carry out a few profiles at different depths. This means that the float's ascent times are known in advance. All the data sets produced are stored in a csv file and transferred by satellite to a data server. This data will then be showcased on NAUTILOS data services through ERDDAP™.

a. Demonstration on seawater pool in France (IFREMER)

- **Specific Objectives**

The demonstration in a seawater pool is an important stage between numerical modelling and the first trials of the float in seawater. This demonstration was specifically designed to allow verification of communication and transmission protocol and the buoyancy of float in a real environment (seawater pool).

- **Platform involved**

The platform concerned is a new generation of profiling float developed by NKE Instrumentation, namely the CTS5 (Fig. 1). This float is fitted with a Seabird CTD that records temperature, conductivity and pressure in the water column at a frequency of 2 s. The silicate sensor is mounted at the base of the float for balance. The float must remain vertical when at the surface and transmitting.

Figure 1: A CTS5 profiling float

Before deploying the CTS5 float in the Mediterranean Sea, we carried out some trials in the IFREMER pool filled with seawater and with a depth of 20 m (Fig. 2).

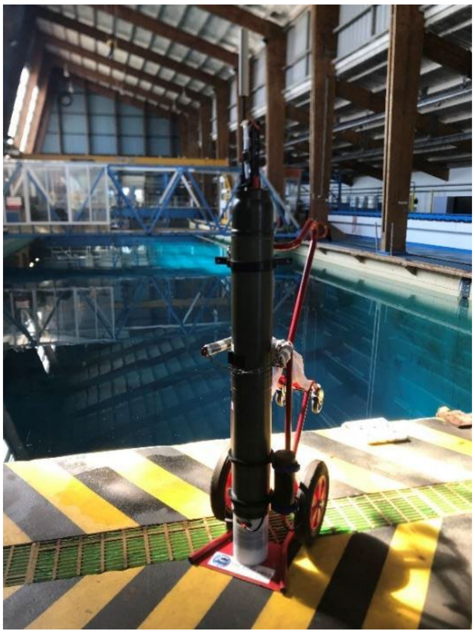


Figure 2: A seawater pool (infrastructure owned by IFREMER)

The objectives of this stage in ST7.4 were to check the overall performance of the sensor (acquisition, measurement, transmission on start-up) and the stability of the float in a quasi-real environment, a seawater pool. The float was deployed in a seawater pool and slid along a cable to a depth of 20m (Fig. 3). A system of links is used to keep the float vertical, as shown in the figure below. The tests consist of several measurement profiles down to a depth of 20 metres.

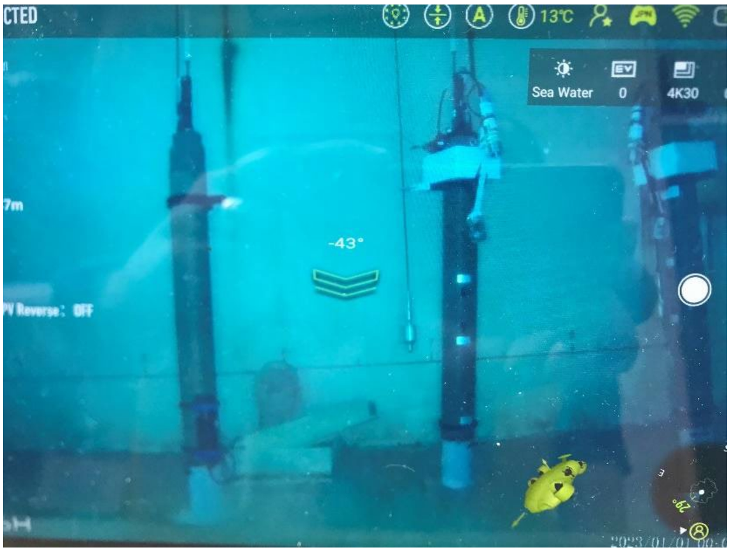


Figure 3: Deployment of silicate float at 20m depth in a seawater pool (IFREMER)

- **Deployment configuration**

The following equipment was installed on the CTS5 profiling float (sn: P50900-17FR002):

- Electrochemical silicate sensor (serial number: 00),
- Seabird CTD (serial number: 41-9081)

The silicate sensor was integrated on the CTS5 float following all the mechanical and electronic integration phases described in D5.5. The silicate sensor was installed at the base of the float near the ballast to keep the float stable and in a vertical position. Moreover, in this position, the complexation cell is not contaminated by any residues floating on the surface. The sensor is positioned upside down. Seawater is pumped to the side of the sensor via a circulator. The measuring chamber is thus renewed and cleaned with each measurement cycle. The sensor is connected to the float via a waterproof SUBCONN cable, so it is powered by the platform and transmits its files to it (Fig. 4).

Figure 4: Integration of silicate sensor on the CTS5 float

The small reservoir seen above the sensor is used to keep the internal membrane moist. Once deployment is programmed, this funnel is removed and replaced by a grid that filters at the seawater circulator inlet.

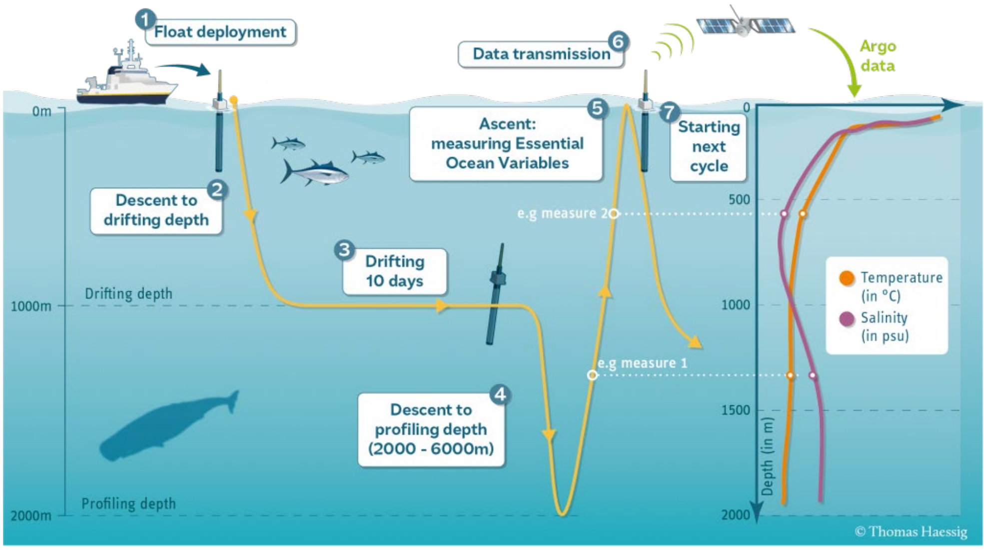
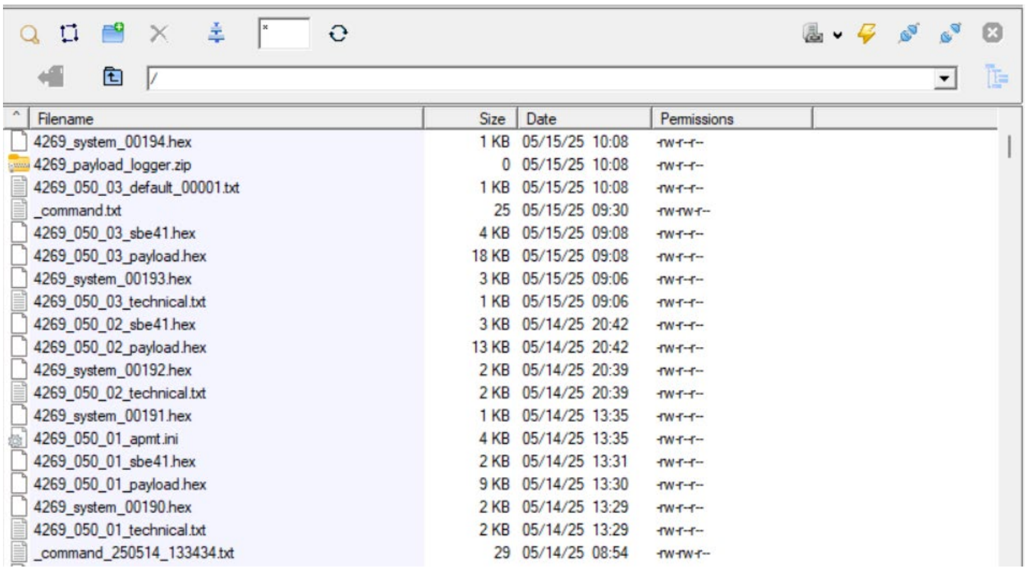


Figure 5: A drawing of What does an Argo float do? (https://argo.ucsd.edu)

In a standard operation, the Argo float mission is carried out on a cycle of 10 days, with most of the float's time spent drifting along with deep ocean currents, followed by taking a series of measurements as it moves back up (profiles) to the ocean surface. As shown in Figure 5, once the CTS5 float is on the surface, it gets its location, often through GPS, and then communicates with a satellite to send its data and receive any new mission instructions. For most of the Argo fleet, this surface interval is between 15 minutes and one hour. This time is necessary for the float to transfer technical, GPS files and other types of data via Iridium satellite and stored on the FTP server (Fig. 6).



Filename	Size	Date	Permissions
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4269_payload_logger.zip	0	05/15/25 10:08	-rw-r--r--
4269_050_03_default_00001.txt	1 KB	05/15/25 10:08	-rw-r--r--
_command.txt	25	05/15/25 09:30	-rw-rw-r--
4269_050_03_sbe41.hex	4 KB	05/15/25 09:08	-rw-r--r--
4269_050_03_payload.hex	18 KB	05/15/25 09:08	-rw-r--r--
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4269_050_01_technical.txt	2 KB	05/14/25 13:29	-rw-r--r--
_command_250514_133434.txt	29	05/14/25 08:54	-rw-rw-r--

Figure 6: Files transmitted by the CTS5 float and stored on the FTP server

After that, the CTS5 float sinks to a drift depth of 1000 meters for about 9 days and then the float sinks to its profile depth of 2000 meters before slowly rising to the surface while measuring conductivity, temperature and pressure. This cycle repeats until the float dies usually 4 – 5 years later. The standard mission was modified and adjusted for pool and seawater testing.

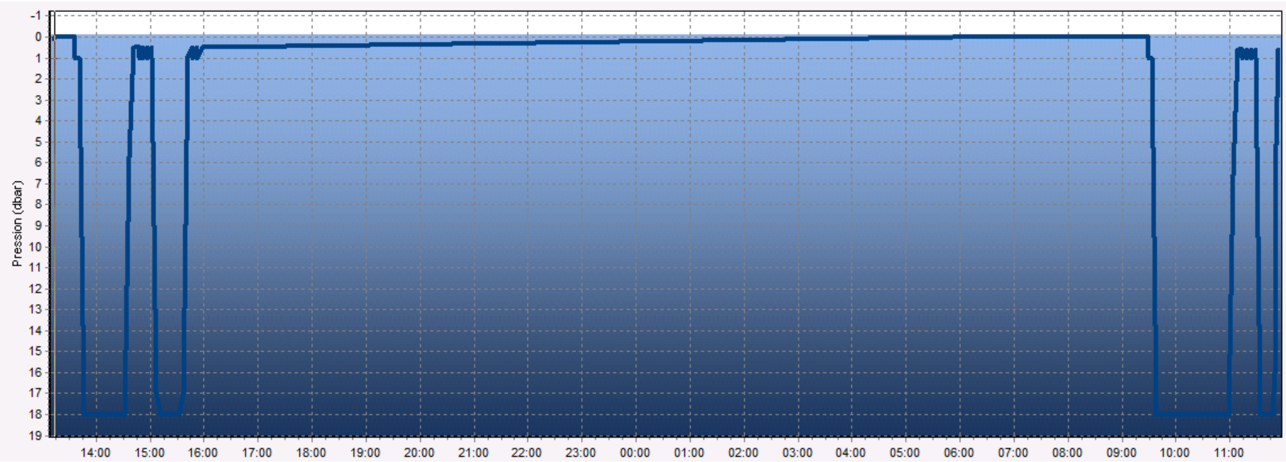


Figure 7: Float's mission in the Ifremer Pool

Regarding pool testing, the float was configured to trigger the silicate sensor measurement cycle at 20 m depth. We configured the silicate sensor for a "pool" mode as shown in Figure 7. As the sensor has an acquisition frequency of one measurement every 10 min, we have programmed silicate measurements during the profiler's parking phases, i.e. 20m depth. The silicate sensor is equipped with a pressure sensor, so we can follow the depth achieved by the sensor. In parallel, a Seabird CTD records with a frequency of 2 seconds during the ascent timing. The CTD can measure a few measurements during the parking phase.

The silicate sensor settings were determined via its own web application using a laptop computer (Fig.8). This configuration enables measurement cell rinsing, electrode cleaning, molybdenum oxidation, complexation and silicate measurement times to be set based on square-wave voltammetry (SWV).

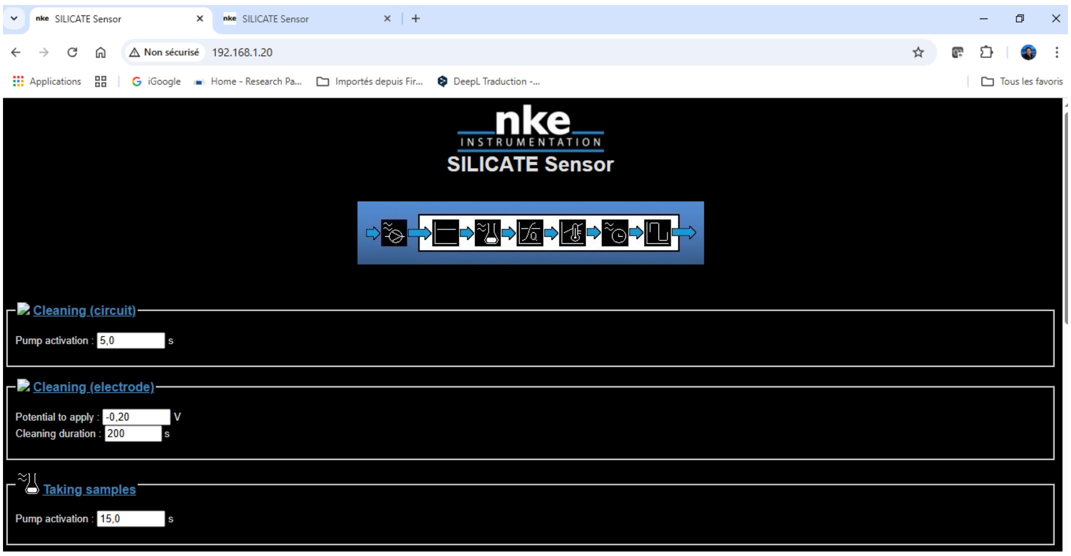


Figure 8: Silicate acquisition settings (web interface) for the demonstration in a seawater pool (IFREMER)

Via this web interface (Fig. 9), it is possible to view the progress of the measurement cycle upstream of sensor integration on the profiler. This tool is particularly useful during sensor calibration, or when checking the sensor after a test campaign.

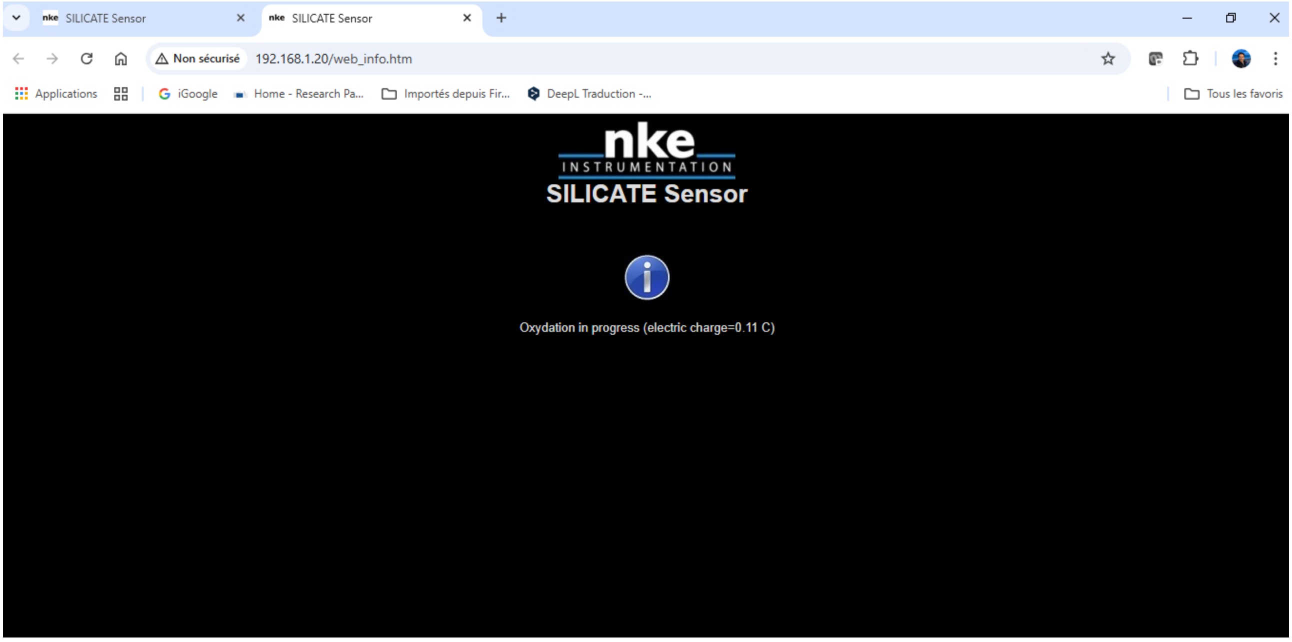


Figure 9: Monitoring the progress of the various silicate sensor actions

A second tool used to communicate with the silicate sensor is the Telnet interface (Fig. 10). We use it to trigger commands such as measurement acquisition, cell pumping and to check various data carried on the sensor. Before installing the sensor on the profiler, we triggered the datalogger: on command to put the sensor in acquisition mode.

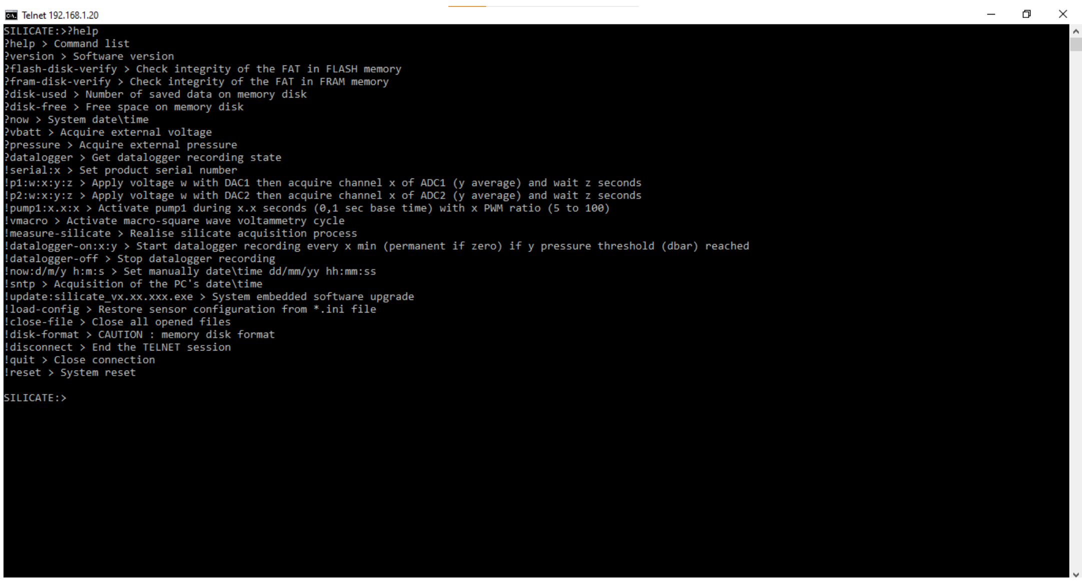


Figure 10: Silicate acquisition settings (Telnet tool) for preparing the silicate sensor

- **Location**

The Ifremer swimming pool is located in the IFREMER centre on the Brest-Iroise technology park in Plouzané near Brest (Fig. 2). The pool is 20 m deep and measures 50 m long.

- **Period of occurrence**

A precalibration of the silicate sensor was carried out with new Certified Reference Materials (CRMs purchased from KANSO CO., LTD., Osaka, Japan) before the seawater trials. Tests in a seawater pool have been very short. Several days were enough to check that the float (buoyancy, communication…) and the silicate sensor was working properly. CNRS-LGC and NKE were in constant contact during this phase. After integration on the ARGO float in May 2024 and testing in a seawater pool in June 2024, the silicate sensor was sent directly to Villefranche-Sur-Mer for its first deployment in the Mediterranean.

- **Maintenance, events/issues occurred and actions taken**

No maintenance was reported during this demonstration stage in a seawater pool. The float fitted with a silicate sensor has successfully completed several profiles and is ready for the next stage: its demonstration in the Mediterranean Sea.

- **Data recovery**

To prepare the NRT data to be implemented during the demonstration phase, some sample data, generated by the CTS5 float were shared with the ETT staff and used to populate the NAUTILOS ERDDAP™ server and test the data transfer to the EMODnet platform.

The data acquired and collected on the float is stored on an FTP server. The data files must first be decrypted and then post-processed by NKE before being shared with ETT staff. In recent weeks, NKE and ETT have finalized the transmission of NRT data provided by the CTS5 float to NAUTILOS services. The CSV (Comma-separated values) data files will be transferred to the ERDDAP™ server via the FTPS (File Transfer Protocol Secure) protocol. Some of the content of the post-processed files is not useful to the ETT team. This information is needed by NKE to check that the silicate sensor is behaving correctly. Initially, only the voltammogram data will be displayed on the EMODnet platform. A future peak recognition tool will be created to generate the actual silicate concentration values directly.

[SQUARE_WAVE_VOLTAMMETRY]					
Starting potential=600.0 mV	Stopping potential=-100.0 mV	Pulse height=20.0 mV	Staircase step size=5.0 mV	Frequency=25.0 Hz	
27/06/24 10:33:35 (40.0 ms)					
U1 (V)	U2 (V)	I1 (µA)	I2 (µA)	Umoy (V)	I2 - I1
0,58	0,6201	-8,283	-4,053	0,60005	4,23
0,5751	0,6152	-8,481	-4,578	0,59515	3,903
0,5702	0,6102	-8,655	-4,659	0,5902	3,996
0,565	0,6052	-8,847	-4,752	0,5851	4,095
0,5602	0,6003	-9,069	-4,869	0,58025	4,2
0,5548	0,5951	-9,3	-4,998	0,57495	4,302
0,5501	0,5902	-9,543	-5,109	0,57015	4,434
0,5452	0,5852	-9,789	-5,25	0,5652	4,539
0,54	0,5801	-10,026	-5,409	0,56005	4,617
0,5351	0,5753	-10,296	-5,544	0,5552	4,752
0,5298	0,5702	-10,539	-5,718	0,55	4,821
0,5252	0,5652	-10,8	-5,892	0,5452	4,908

Figure 11: Examples of data files produced by silicate sensor

Figure 11 shows a pre-agreed data format with the measured parameters and their respective quality flags. A diagram of the general data and metadata flow between data provider (NKE) and the NAUTILOS ERDDAP™ server (managed by ETT) is shown in Figure 12.

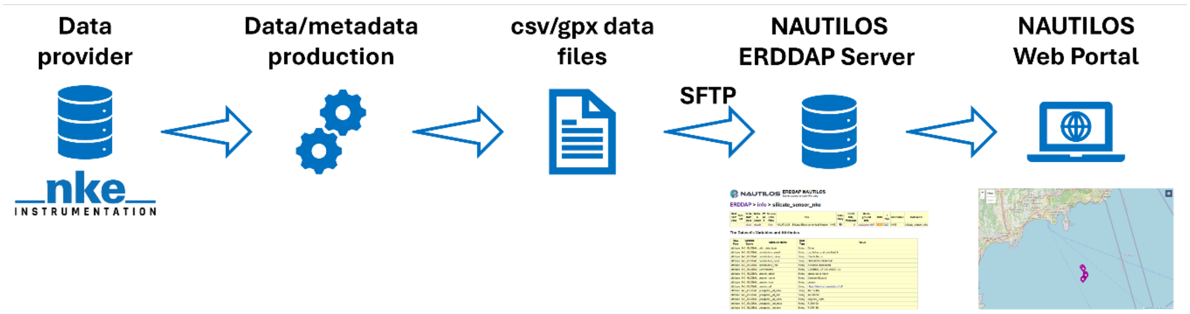


Figure 12: General data flow between the data provider (NKE) and NAUTILOS ERDDAP server (to be modified)

Then an automatic service gets metadata from the data from the CSV file, merging them in a single Network Common Data Form (NetCDF) file (one file for each data package). The NetCDF files are uploaded and made available through the ERDDAP™ server (Fig. 13).

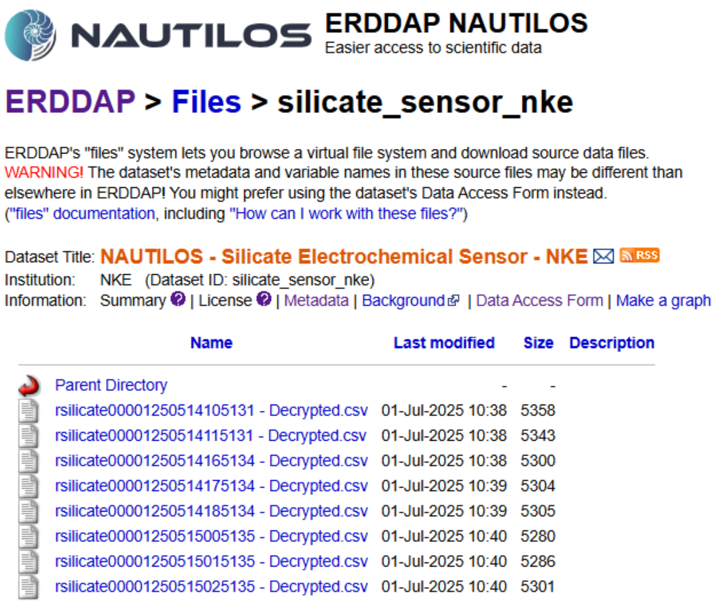


Figure 13: NetCDF files generated and uploaded to the NAUTILOS ERDDAP

b. First Demonstration on ARGO float in the Mediterranean Sea (CNRS-LGC)

- **Specific Objectives**

This first demonstration was specifically designed to allow verification of sensor robustness, acquisition measurement, silicate measurement in real environment up to 1000 m of depth and communication and transmission protocol and to showcase data collected in near real-time on NAUTILOS data portals.

- **Platform involved**

The platform concerned is a new generation of profiling float developed by NKE Instrumentation, namely the CTS5 (Fig. 1). This float is fitted with a CTD Seabird that records temperature, conductivity and pressure in the water column at a frequency of 2 s. The silicate sensor is mounted at the base of the float for balance. Indeed, the float must remain vertical when at the surface and transmitting.

Figure 14: A Sagitta III research vessel (owned by CNRS-DT INSU)

To deploy the CTS5 float, a research CNRS-DT INSU boat based in the port of Villefranche-Sur-Mer was selected to participate in NAUTILOS demonstrations on the ARGO float in ST7.4. The vessel, SAGITTA III, is equipped for inshore fishing, operates up to 20 miles off the coast, mainly in the Villefranche-Sur-Mer roadstead and Bouée Boussole areas (Fig. 14). It is mobilized to support various observation, research and teaching activities in marine biology and ecology, biogeochemistry, seismics and oceanography. This CNRS-DT INSU boat is equipped with a fishing winch, a hydrology winch and an electrotransport winch, making it easy to launch profilers. The profiler was installed on its stern and can be easily launched by means of a cable and an adapted release system (Fig. 15).

Figure 15: Silicate profiling float fixing system for launching

- **Deployment configuration**

Please refer to section II.a - Deployment configuration. The CTS5 profiling float (sn: P50900-17FR002) is configured identically with an electrochemical sensor (serial number: 00) and a Seabird CTD (serial number: 41-9081). The float is configured to trigger the silicate sensor measurement cycle at different depths. As the sensor has an acquisition frequency of one measurement every 10 min, we have programmed silicate measurements during the profiler's descent phases (Fig. 16). For the demonstration, we initially set 3 depths: 250m, 350m and 800m.

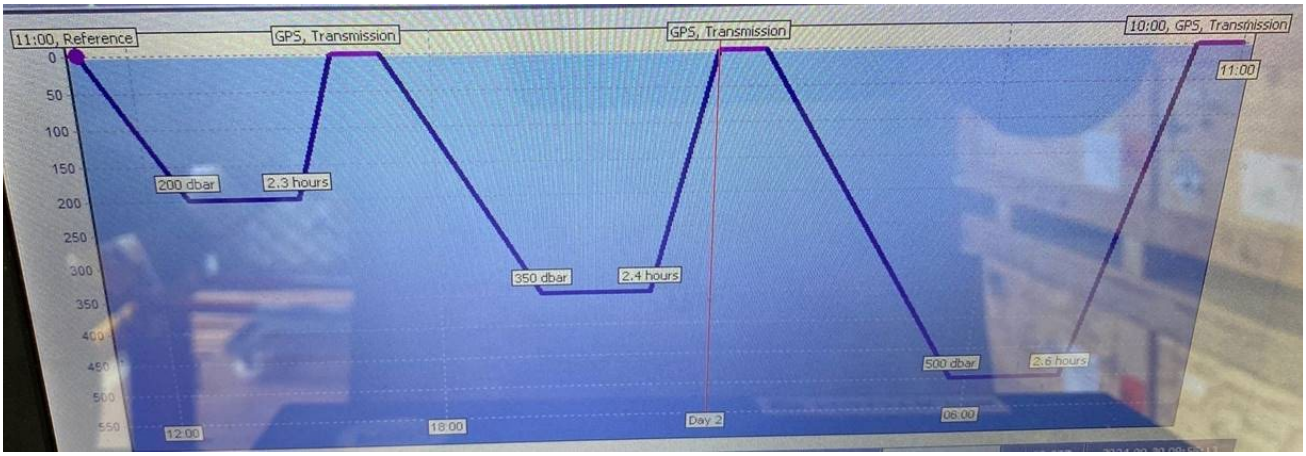


Figure 16: Mission of the float in the Mediterranean Sea in July 2024

In parallel, the CTD Seabird records with a frequency of 2 seconds during the ascent timing. The CTD can measure a few measurements during the parking phase. The aim was to program a short float mission, so that the float could be recovered close to shore. If the float had strayed too far from shore, we would have sent a command to the float to continue its mission.

As in the Ifremer pool, the silicate sensor settings were determined via its own web application using a laptop computer. This configuration enables measurement cell rinsing, electrode cleaning, molybdenum oxidation, complexation and silicate measurement times to be set based on square-wave voltammetry (SWV). Via this web interface, it is possible to view the progress of the measurement cycle upstream of sensor integration on the profiler. This tool is particularly useful during sensor calibration, or when checking the sensor after a test campaign.

- **Location**

The SAGITTA III is routinely operating in the Mediterranean Sea and more specifically off Villefranche-Sur-Mer. The Mediterranean is renowned for its deep waters. From the port of Villefranche-Sur-Mer, just a few miles off the coast, it is very easy to reach depths of 2,000 metres (Fig. 17).

Figure 17: Location of float demonstration in the Mediterranean Sea, South of France

In this part of the Mediterranean, the currents flow mainly in an East-West direction. These currents are named the Northern or Ligurian Current, which originates off Palermo, in the south of the Tyrrhenian Sea (Fig. 18). It heads towards the Italian coast to the north, approaching the Bay of Naples and then the Gulf of Genoa, in the Ligurian region that gives it its name. It then heads westwards and, off Monaco, joins the currents flowing up from the Corsican coast.

As a result, knowledge of this current makes it easier to track a float deployed off the coast of Monaco. Off the coast of Nice is the DYFAMED observation buoy, which is helping to improve our understanding of the mesoscale variability of hydrological and biogeochemical processes in the Ligurian Sea. This buoy is used to monitor the impact of climate and anthropogenic forcing on the evolution of water masses in relation to the formation of deep waters and the propagation of masses from the eastern basin, on biological production and the export of associated matter (nutrients, major elements C, N, Si), acidification and the sequestration of anthropogenic carbon, biodiversity and the variability of atmospheric inputs on the biological CO_2 pump.

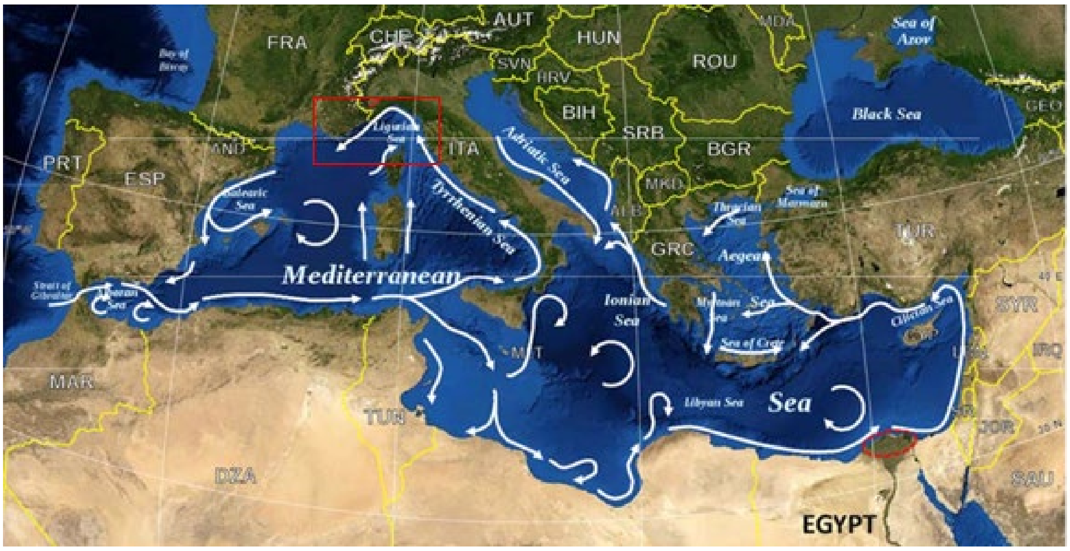


Figure 18: Ligurian Sea in the Mediterranean Sea

- **Period of occurrence**

The initial demonstration was scheduled for 4 July 2024, but a GPS issue was encountered, and the float was sent to NKE for maintenance in mid-July 2024. The antenna was replaced, and new transmission tests were carried out successfully. The fault has been reported. CNRS-LEGOS, NKE and the ship's captain were in constant contact during this phase to plan a new demonstration. A new mission was planned for around 9 October 2024. The float was shipped to Villefranche-Sur-Mer on 4 October 2024. For its part, NKE prepared the silicate sensor in the laboratory by carrying out a series of molybdenum oxidation tests to prevent the electrodes from becoming passivated during the campaign and preventing the product from functioning. However, we had to complete a request for a reservation at sea because we didn't have any during this period. The new campaign on 9 October 2024 was cancelled and postponed due to poor weather conditions (1.7 m swell). As the SAGITTA III program was full until 11 December 2024 and work was underway in Toulon, our demonstration at sea was postponed until 2025. NKE took the opportunity to run the silicate sensor again in a hyperbaric chamber at 100 bars. The captain of the SAGITTA II suggested that we deploy the system in March 2025, without being sure of the weather conditions. At the beginning of March 2025, conditions turned out to be poor. The demonstration had to be postponed to 19/20 and 21 March 2025. With no response from SAGITTA III and despite several reminders, we finally obtained a slot for May 2025. We went down to Villefranche-Sur-Mer to install the silicate sensor on the CTS5 float on 28 April 2025. On site, the sea trip was cancelled because the SAGITTA III was unavailable due to last-minute testing priorities. As a result, the float fitted with the silicate sensor remained at the Villefranche-Sur-Mer facility.

Given the difficulties in finding a slot at sea, we opted for a short campaign solution to allow for easier recovery of the float in the Mediterranean Sea. Ultimately, the float was deployed in mid-May 2025, which means there was an 11-month wait before it could be deployed.

- **Maintenance, events/issues occurred and actions taken**

Following a problem with the transmission of the GPS point at the time of the autotest (full mode) before the float was launched on the same day at sea, we had to stop the mission. Despite several attempts, we forced the GPS synchronization via a Telnet command. This solution failed, highlighting an antenna issue. Without the float's GPS position, it would have been impossible for us to recover it at sea. It was therefore sent back to NKE for evaluation. After evaluation, the float showed a defect in its antenna. It is possible that the antenna suffered shocks during transport between Hennebont and Villefranche-Sur-Mer, which may have damaged it. The float was reassembled at NKE and retested in the tube. We have rescheduled a new demonstration in the Mediterranean Sea.

- **Data recovery**

The initial mission on 4 July 2024 failed, so no data was recovered. As shown in the figure below, the "check GPS" setting displays an error message (Fig. 19).

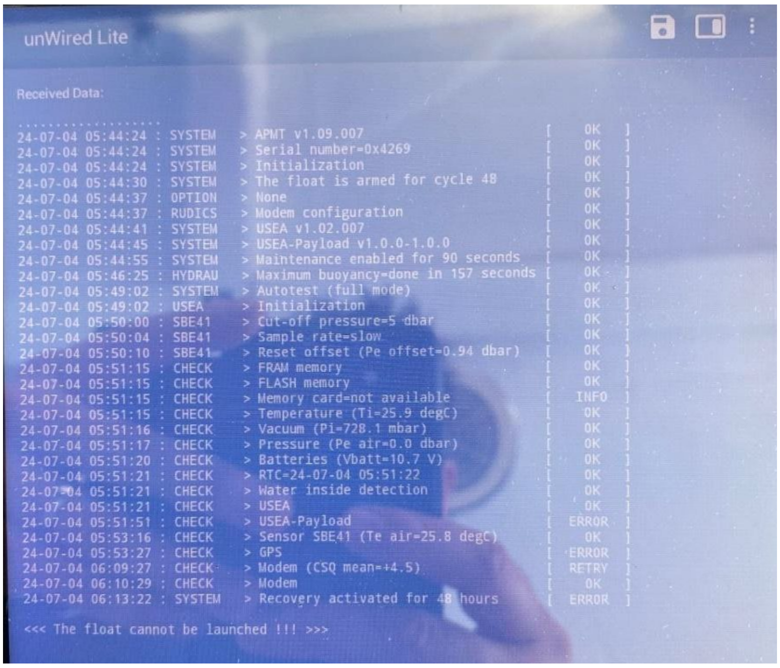


Figure 19: Failure of the CTS5 float self-test

c. Second Demonstration on ARGO float in the Mediterranean Sea (CNRS-LGC)

- **Specific Objectives**

This first demonstration was specifically designed to allow verification of sensor robustness, acquisition measurement, silicate measurement in real environment up to 1000 m of depth and communication and transmission protocol and to showcase data collected in near real-time on NAUTILOS data portals.

- **Platform involved**

The platform in question is similar to the one used during the first demonstration. Please refer to the section II.b - platform involved.

- **Deployment configuration**

Please refer to the section II.b - Deployment configuration.

- **Location**

Please refer to the section II.b - Location.

- **Period of occurrence**

We went down to Villefranche-Sur-Mer to install the silicate sensor on the CTS5 float on 28 April 2025. The float fitted with the silicate sensor was deployed the 14th of May 2025 at 10 am (Universal Time) for a period of 24 hours. The float was recovered the 15th of May after having carried out 3 profiles between 250 and 560 m. Once recovered, the silicate sensor was sent to NKE Instrumentation for post-calibration and maintenance.

- **Maintenance, events/issues occurred and actions taken**

The deployment of the CTS5 float equipped with a silicate sensor was successful. However, the recovery was more complicated than expected. A bracket was broken during the recovery. Fortunately, there was no damage to the float and the operators (Fig. 20).

Figure 20: Fixation mounted on the float's body

- **Data recovery**

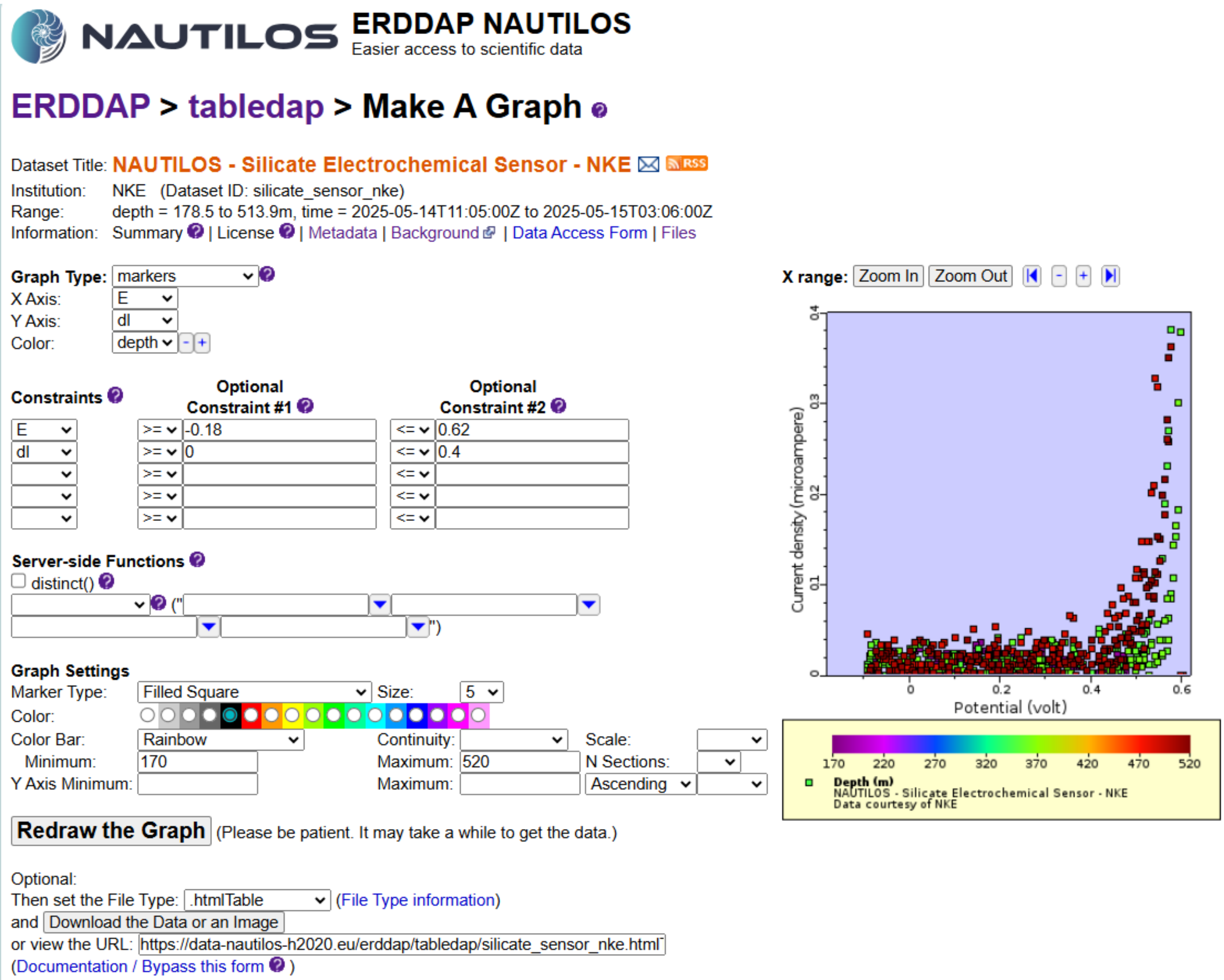
To progress the NRT data to be implemented during the demonstration phase, some new sample data, generated by the CTS5 float were shared with the ETT staff and used to populate the NAUTILOS ERDDAP™ server managed by ETT and Data Portal managed by CNR-ISTI.

The data acquired and collected on the float is stored on an FTP server. The data files must first be decrypted and then post-processed by NKE before being shared with ETT staff. In a few weeks' time, NKE and CNR will finalise the transmission of NRT data provided by the CTS5 float to NAUTILOS services. The CSV (Comma-separated values) data files will be transferred to the ERDDAP™ server via the FTPS (File Transfer Protocol Secure) protocol. Some of the content of the post-processed files is not useful to the ETT team. This information is needed by NKE to check that the silicate sensor is behaving correctly. Initially, only the voltammogram data will be displayed on the NAUTILOS ERDDAP™ (Fig. 21) and Data Portal (Fig. 22). A future peak recognition tool will be created to generate the actual silicate concentration values directly.

Data are available on ERDDAP™ at the following links:

- Electrochemical silicate sensor: https://data-nautilos-h2020.eu/erddap/tabledap/silicate_sensor_NKE.html
- CTD Seabird: https://data-nautilos-h2020.eu/erddap/tabledap/seabird_ctd_NKE.html

a)



b)

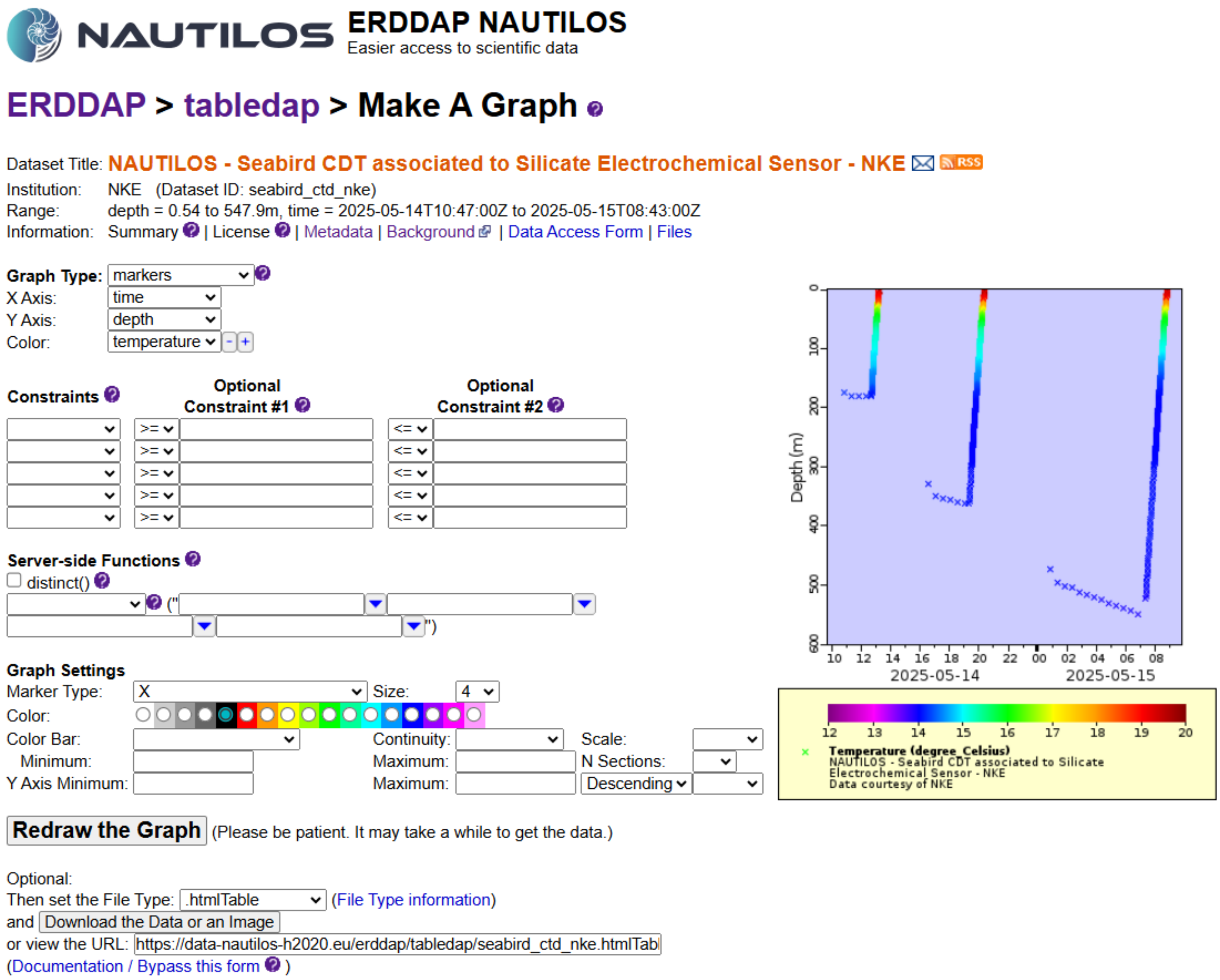


Figure 21: Datasets available on NAUTILOS ERDDAP™: a) Silicate sensor, b) CTD Seabird

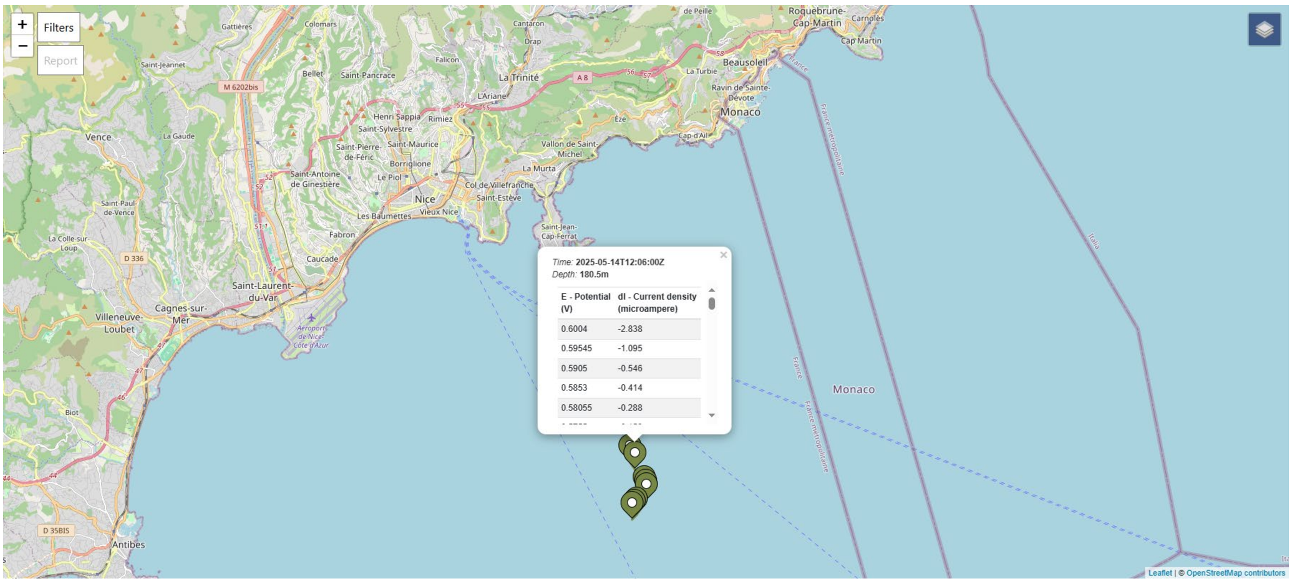


Figure 22: Visualisation of silicate sensor dataset on NAUTILOS Data Portal

3. Results – Comments – Conclusions

- **Results**

The float was assembled and prepared at NKE. A battery of tests was carried out in our 6 m tubes (Fig. 23). As these tubes are filled with fresh water, it is not possible to analyse the voltammograms recorded by the silicate sensor. However, this step was necessary to check the acquisition, communication and transmission chains between the silicate sensor and the float.

Figure 23: Tests of silicate probe in a freshwater tube (6m of depth)

Once the buoy was equipped with the silicate sensor at sea, we proceeded with a test phase in the seawater pool at Ifremer. Since the buoy was weighted in freshwater at NKE, it was necessary to validate the buoyancy parameters in the Ifremer basin filled with seawater. Figure 24 shows the mission carried out by the CTS5 float equipped with silicate sensor in the IFREMER basin. The float carried out 4 profiles down to 20m.

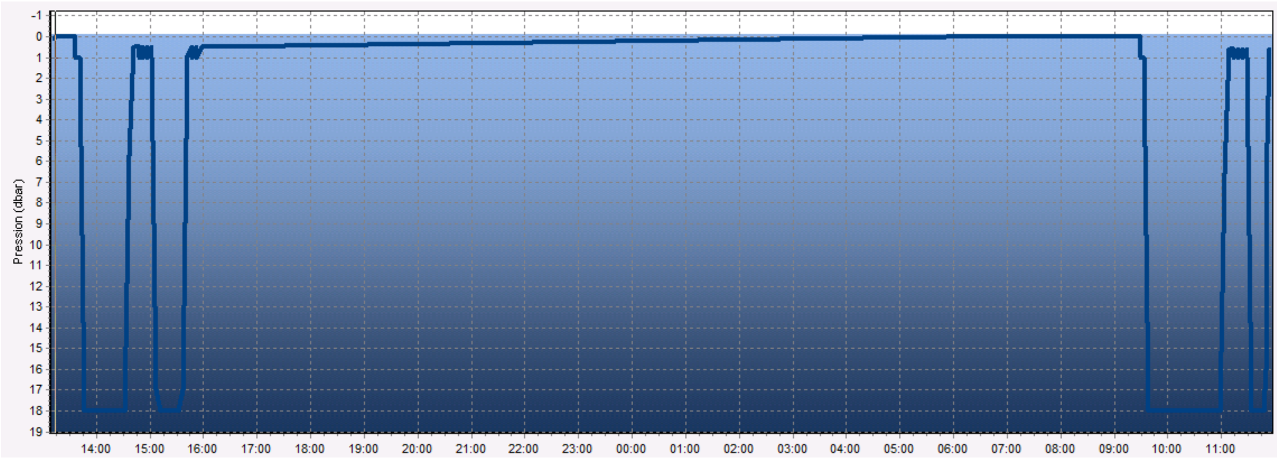


Figure 24: Float's mission in the Ifremer Pool

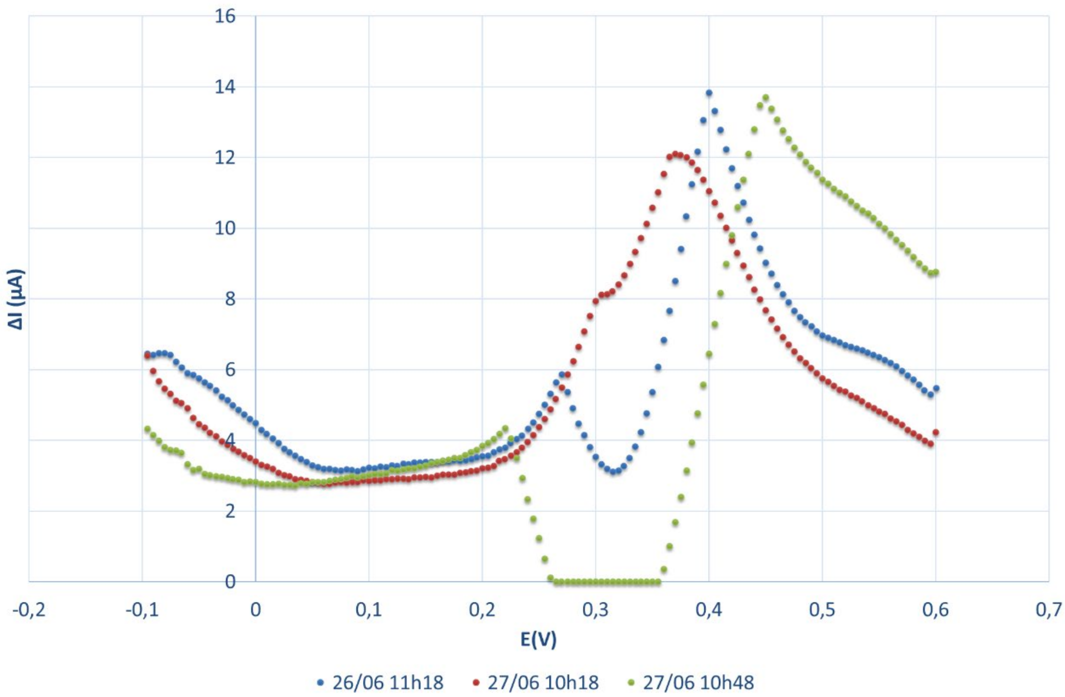


Figure 25: First voltammograms measured at 20 m deep in the IFREMER pool

The silicate sensor recorded three voltammograms (ΔI = f(E)) corresponding to the three descents made by the ARGO float in the Ifremer seawater basin. The aim was to ensure that the measurement cycles were working correctly. No samples were taken from the basin to analyse the silicate concentration. However, the voltammograms recorded show peaks of 12 and 13 µA at potentials of 0.35V (Fig. 25). These could indicate the presence of silicate in the basin. The zero values on the third voltammogram could indicate the presence of bubbles in the measuring cell. These could cause short circuits during recording.

Regarding the first campaign in the Mediterranean Sea, no sensor data was recorded because the float was not deployed due to antenna problems. Without transmitting the GPS position, the profiler could have been lost. A new mission was rescheduled.

The second demonstration campaign for the silicate sensor was relatively short (24 hours) due to boat availability constraints in the Mediterranean Sea. To prevent the float from drifting too far from the coast, we decided to deploy it less than 10 miles from the shore. This mission was carried out at an accelerated pace, as this type of float usually takes a profile every 10 days. We sped up the process by taking three profiles at 250 m, 350 m and 530 m in 24 hours. The pressure trace from the pressure sensor of the SBE probe mounted on the float is shown in Figure 26.

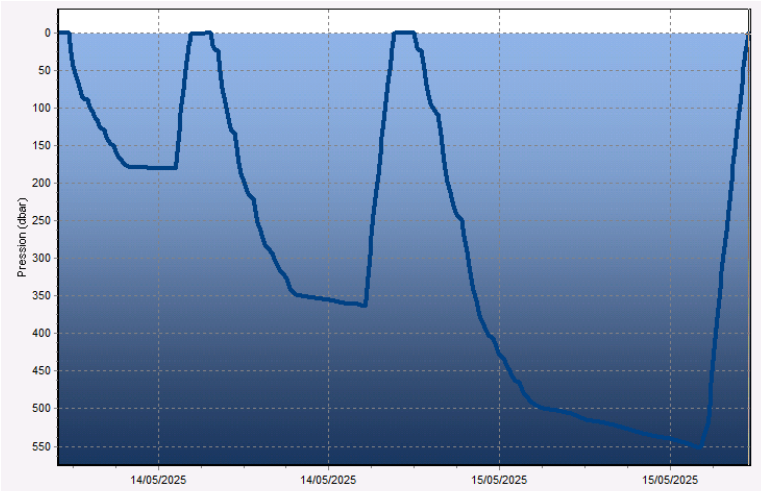


Figure 26: Mission of the CTS5 float after 24h in the Mediterranean Sea

From 14 May 2025 to 15 May 2025, the silicate sensor recorded three readings during the stationary phase in the Mediterranean Sea. The float recorded three profiles at different depths. The float also transmitted GPS positions after each ascent. Figure 27 below shows the trajectory of the float off the coast of Villefranche-Sur-Mer. It follows the Ligurian current from east to west.

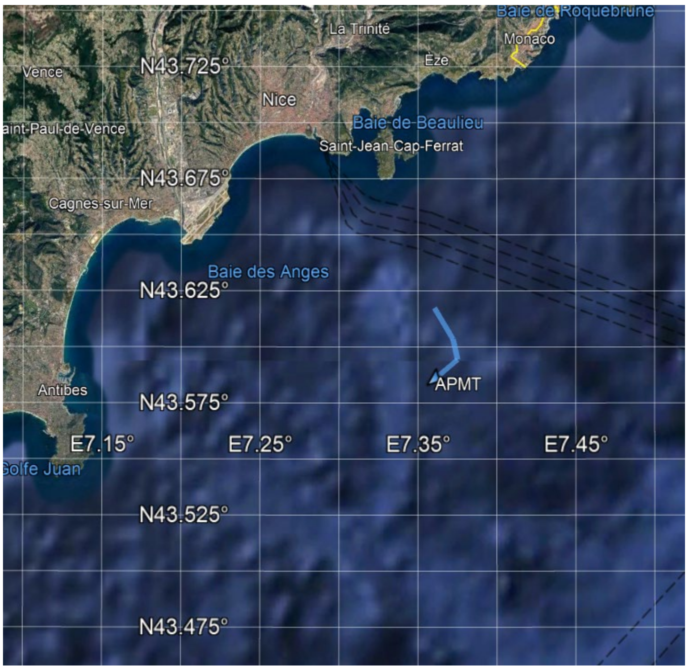


Figure 27: Trajectory of the silicate float in the Mediterranean Sea

After decoding the files, we plotted the voltammograms of the silicate sensor at each parking depth. At the same time, we plotted the temperature, salinity and pressure data provided by the SBE41. The silicate sensor encountered water temperatures ranging from 13.5°C at depth to 20°C at the surface (Fig. 30), and salinity ranging from 37.2 psu at the surface to 38.7 psu at depth (Fig. 31). The current values displayed on the voltammogram were relatively low (<0.05 µA), indicating very low silicate concentrations (Fig. 29). For comparison, the Boussole buoy off Nice measures silicate concentrations of less than 5 µmol/L between 0 and 800 m depth. Due to difficulties in finding a time slot with the SAGITTA III teams, we were unable to take reference samples at the time of launching and on the water column. As compared with the calibration curve of the silicate sensor (Fig. 28), the silicate values are smaller than 2 µmol/L).

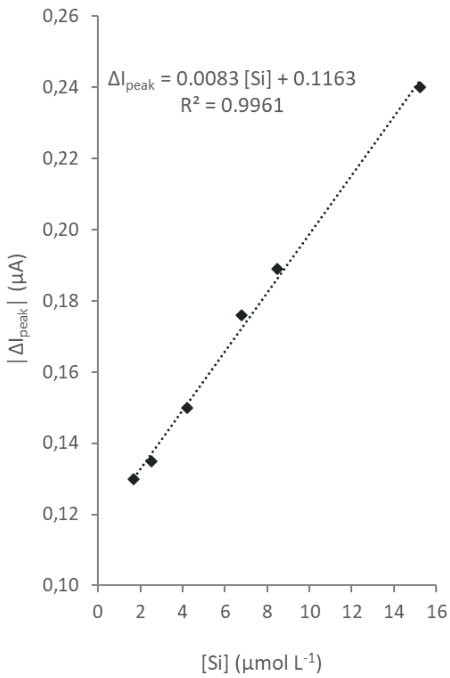


Figure 28: Calibration plots of silicate in situ electrochemical sensor

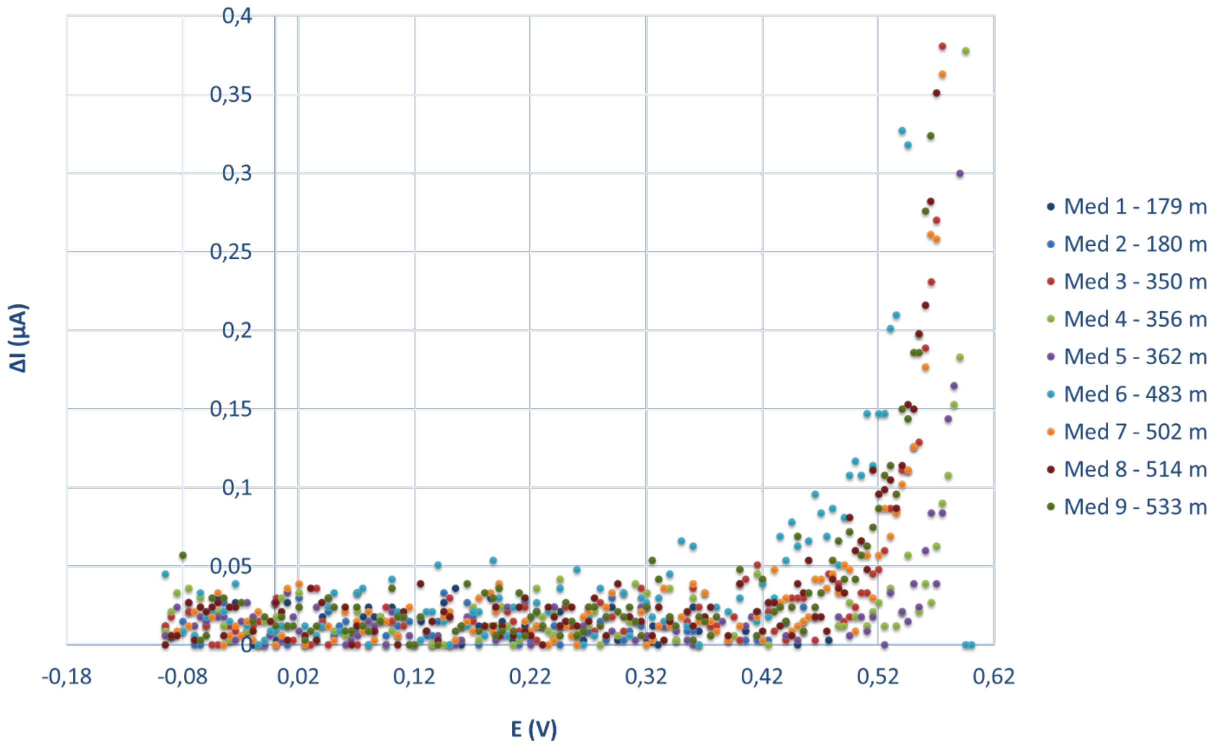


Figure 29: Voltammograms recorded by the silicate sensor in the Mediterranean Sea

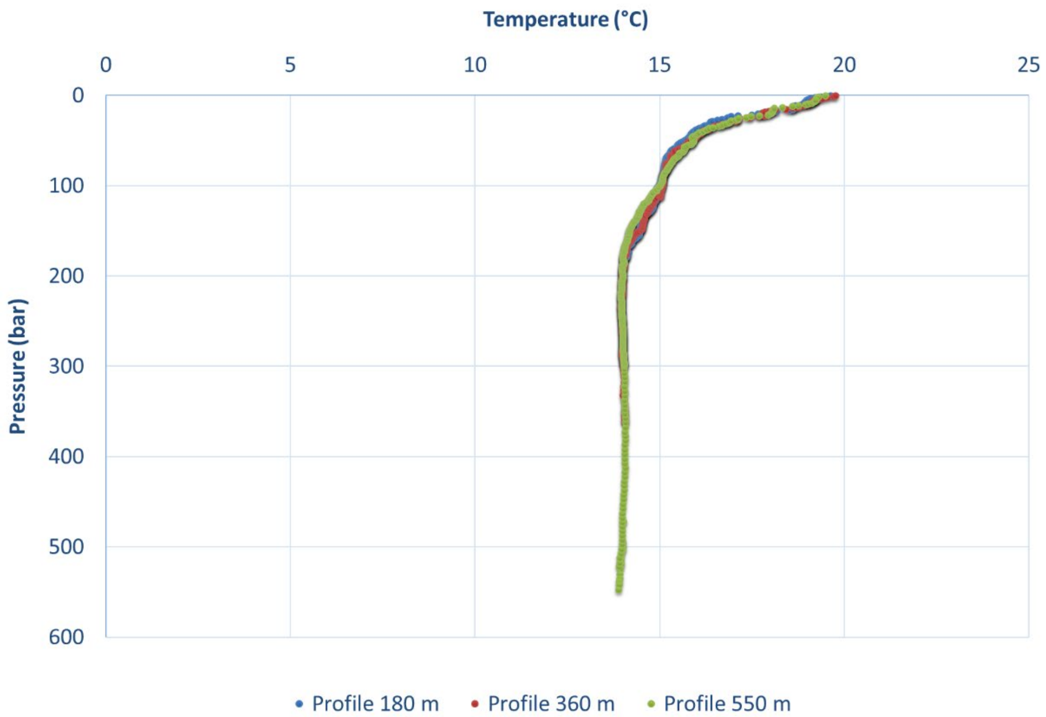


Figure 30: Temperature measurements recorded by the SBE41 sensor as function of in situ pressure in the Mediterranean Sea

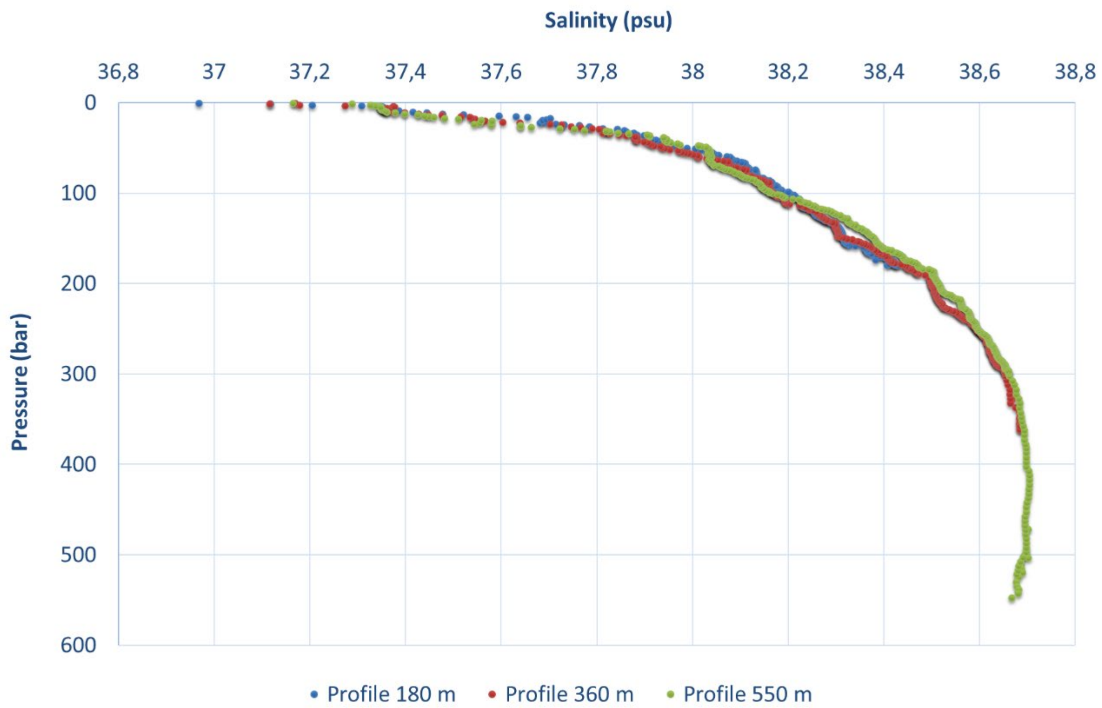


Figure 31: Salinity measurements recorded by the SBE41 sensor as function of in situ pressure in the Mediterranean Sea

This demonstration in the Mediterranean Sea validated the integration of the silicate sensor on the CTS5 profiler. As for the next steps for the sensor, it will be necessary to continue evaluating the sensor at pressures between 0 and 100 bar in silicate-rich waters to refine the fluid circulation in the measuring chamber and limit the stagnation of micro air bubbles trapped in the cell where complexation takes place.

- **TRL achievement level and project SOP**

Based on the demonstration phase, communication between the silicate sensor and the ARGO float was successfully tested. The sensor's integrated software needs to be updated to enable it to provide silicate concentration data. Due to these future improvements, the declared technology readiness level (TRL) is 8 at this stage.

- **Future plans and Recommendations**

Over the coming months NKE staff will continue to work on industrialising the product and will target new measurement campaigns with the silicate sensor in the ocean. These will be repeated to assess the reproducibility of the measurements on several serial sensors. Firstly, NKE will recover the silicate sensor and test it in the laboratory to ensure that nothing has interfered with the pressure measurements in the Mediterranean Sea. The silicate sensor will be tested in a seawater bath and several measurement cycles will be run. Post-calibration could be envisaged for future product demonstrations. This demonstration will be the first and not the last.

NKE would also like to generate synergies with the silicate scientific community and launch new projects with the aim of deploying the sensor in different seawater matrices around the world. Indeed, the Pacific is a part of the ocean where concentrations are the highest. NAUTILOS has already reached a depth of 500 m and we hope to be able to go down to 1000 m in the near future.

NKE would also like to equip itself with a hyperbaric chamber filled with seawater to qualify the silicate sensor under pressure and in real seawater so that the measurement chain is possible.

During the campaign in the Mediterranean Sea, we found that the pumping circuit could interfere with the SWV measurements following the complexation of the silicates with the molybdenum. There will be recommendations for the next time the sensor is put in the water. To prime the water circulator, make sure the cell is full of water and not trapped in bubbles.

III. Ethical Considerations

1. Data Protection

Nothing intrinsic in stored data unless the user tags free text with personal/confidential data.

2. Environmental Protection

The silicate sensor is enclosed in a waterproof case and is not considered hazardous. The waterproof housing provides an additional barrier to help isolate these materials from the marine environment. This deployment includes planned recovery, further minimizing environmental risk. In case of an accidental loss during drifting, this product could cause some environmental damage and release small quantities of materials such as copper, lithium, plastics, and antifouling substances when left to degrade on the seafloor. This product collects scientific data using non-destructive methods, therefore is considered a low impact demonstration.

3. Health and Safety

3.1 Dangerous goods classification

Silicate sensor is not considered a hazardous product as such provided it is used under normal operating conditions, i.e. in accordance with the manufacturer's recommendations as given in the user manual or similar documents.

3.2 Battery safety

The silicate sensor can either be powered by an external power supply equipped with lithium batteries (reference: 3.6 V primary lithium-thionyl chloride Li-$SOCl_2$) prohibited for transport (class 9) in the case of stand-alone probes or via an external platform (the profiling float, in our case). Li-$SOCl_2$ batteries are sealed devices that are not hazardous when used under normal conditions in accordance with the manufacturer's recommendations. Under normal use, the integrity of the battery is maintained and the active components within it are not exposed to the outside world. Once inserted into the waterproof housing, the battery is not subject to mechanical abuse. They are used within acceptable thermal ranges.

3.3 Pressure housings

The silicate sensor is subjected to external pressures induced by the marine environment. The waterproof housing is at atmospheric pressure. The enclosure is not dangerous as such. To be compliant for deployment at sea, silicate sensors must meet the AFNOR standard: NF X10-812. The latter is a document devoted to the tests to be performed on oceanographic equipment immersed in a marine environment.

3.4 RoHS

All components selected for the system are RoHS compliant

4. Protection of Marine Life

The silicate sensor is a passive device that does not emit signals, vibrations, or light, and thus does not disturb marine organisms. Its brief deployment and planned recovery ensure minimal interaction with the surrounding ecosystem. Given its design and operational profile, the sensor poses no direct threat to marine life during this demonstration activity.

5. Dual Use Potential

Not applicable.

IV. Summary

Demonstrations carried out as part of Task 7.4 contributed mainly to NAUTILOS Specific Objectives SO2 (Develop and demonstrate improved observing systems in the open ocean and deep-sea environments).

The ST7.4 demonstrations on ARGO floats involving the silicate prototype developed by NKE and CNRS-LGC in ST4.2, were carried out in May 2025 after some validations in IFREMER's seawater pool. The silicate sensor was installed on the ARGO float. The aim of the demonstrations was to evaluate the performance and reliability of the sensor in real conditions at depth. The first campaigns at depths of more than 500 m were validated with the sensor. The technical challenges encountered led to recommendations for the maintenance of the silicate sensor and the preferred operating conditions. Overall, the silicate sensor demonstrated its stability and resilience, and thanks to the demonstration phase, the technological maturity level of the systems has increased to 8. Near real-time data transmission from the ARGO float to an FTP server has been achieved. Real-time data transmission to central repositories is ongoing. The silicate sensor needs to develop a peak recognition tool to provide silicate concentration data (µmol/L). Demonstrations in a pressurised environment will also need to be continued in order to validate the transmission stages.

Appendix 1: References and Related Documents

ID	Reference or Related Document	Source or Link/Location
1	Barus et al., 2021	https://www.sciencedirect.com/science/article/pii/S0925400521002732#fig0025
2		