EuroGO-SHIP
Enhancing ocean observations

D3.2. Report on pilot activities relevant to shared facilities

May 2024

Co-funded by the European Union

UK Research and Innovation

This work was funded by the European Union under grant agreement no. 101094690 (EuroGO-SHIP) and UK Research and Innovation (UKRI) under the UK government's Horizon Europe funding guarantee [grant number 10051458, 10068242, 10068528]. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or European Research Executive Agency. Neither the European Union nor the granting authority can be held responsible for them.

About this document

Title	D3.2. Report on pilot activities relevant to shared facilities
Work Package	WP3, Concept Demonstration
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Due Date	31.05.2024, M18
Submission Date	DD.MM.YYYY
Version	**1.0**

Dissemination Level

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EuroGO-SHIP: Developing a Research Infrastructure Concept to Support European Hydrography is a Research and Innovation action (RIA) funded by the Horizon Europe Work programme topics addressed: HORIZON-INFRA-2022-DEV-01-01– Research infrastructure concept development. Start date: 01 December 2022. End date: 30 November 2025.

EuroGO-SHIP is co-funded by the European Union, Horizon Europe Funding Programme for research and innovation under grant agreement No. 101094690 and by UK Research and Innovation

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1. Introduction

The EuroGO-SHIP project aims to establish a European Research Infrastructure (RI) for hydrography, which is essential for understanding the ocean's role in climate, ecosystems, and human activities. However, the current hydrographic observation system in Europe is fragmented, inefficient, and insufficient to meet the growing demands of science and society. Therefore, there is a need for a coordinated, harmonized, and sustainable RI that can provide high-quality, reliable, and interoperable hydrographic data and related services to the European and global community.

The EuroGO-SHIP project has four main objectives:

- To develop a governance and business model for the European RI for ship-based hydrography;
- To design and implement shared facilities and services that can enhance the efficiency and quality of hydrographic and other ship-based observations;
- To demonstrate the principles and operations of the European RI for hydrography through pilot activities;
- To engage and network with relevant stakeholders and users of hydrographic data and related services.

This deliverable, D3.2, reports on the pilot activities relevant to shared facilities, which are one of the key components of the European RI for hydrography. Shared facilities are resources and capabilities that can be accessed and used by the network of hydrographic observers, such as specialized analyses, reference materials (RM), training, software, best practices, and equipment pool. The purpose of this deliverable is to present and evaluate the results of the pilot activities that have been carried out within Work Package 3 (WP3) to test and demonstrate the shared facilities concept and its benefits for the hydrographic community.

1.1. Background and context of the deliverable

The concept of shared facilities is based on the idea that sharing resources and capabilities among the network of hydrographic observers can significantly improve the quality, efficiency, and impact of hydrographic observations. Shared facilities can also foster collaboration, innovation, and standardization among the hydrographic community, as well as reduce costs and duplication of efforts. This concept paves the way for a more inclusive and democratic approach to hydrography, acknowledging the diverse economic landscapes across Europe. It ensures that countries with limited resources can also contribute to and benefit from the collective advancements in hydrographic science. Aligned with the vision and mission of the EuroGO-SHIP project, the shared facilities concept aims to establish a European RI for hydrography that can provide high-quality, reliable, and interoperable hydrographic data and services, not only to the European community but also on a global scale, democratizing access to oceanographic knowledge.

The shared facilities concept was initially proposed and scoped in WP2, which is responsible for developing the statement of requirement for the European RI for hydrography. WP2 identified six categories of shared facilities that can address the needs and requirements of the network of hydrographic observers, namely:

- Capability: associated with specialized analyses and instrumentation;
- Reference materials: for enabling quality control to produce high-quality chemical analyses;
- Training: including virtual, in land-based laboratories and at sea;
- Software: for primary quality control of observational data;
- Best practice: for all aspects of observing and data handling, including cruise logistics;
- Investigating the opportunity of a European Marine Equipment Pool: for sharing equipment that is otherwise unavailable.

WP2 will also define the objectives, functions, and expected outcomes of each shared facility category, as well as the potential supply and demand models for their provision and access. The preliminary results of WP2 were reported in Deliverable 2.1, which also provided a roadmap for the development and implementation of the shared facilities within the EuroGO-SHIP project.

WP3, which is responsible for demonstrating the principles and operations of the European RI for hydrography through the pilot activities, took the lead in developing and implementing the shared facilities concept in practice. WP3 designed and is executing three pilot activities that are relevant to three shared facility categories, namely:

- Salinity best practice: to evaluate the effect of different salinometers and lab conditions on consistency and stability of bottle salinity measurements, and to update the salinity best practice documentation accordingly;
- Nutrient sample storage: to compare the impact of long-term methods of nutrient sample storage, such as freezing and pasteurization, and to recommend the best practice and fit-for-purpose sample storage method;
- Carbon secondary standards: to produce and distribute a batch of reference seawater for carbon system variables, and to establish a roadmap for creating a European hub for carbon secondary standards.

The pilot activities involved several partners from the EuroGO-SHIP consortium and took advantage of existing or planned cruises in different regions of the European seas, such as the North Atlantic, the Mediterranean and Black Seas. The pilot activities aimed to test and demonstrate the shared facilities concept and its benefits for the hydrographic community, as well as to provide feedback and recommendations for its implementation and expansion.

1.2. Objectives and scope of the deliverable

The main objective of this deliverable is to report on the results and recommendations of the pilot activities relevant to shared facilities that have been carried out within WP3. Its specific objectives are:

- To describe the rationale, methodology, and data sources of each pilot activity;
- To present a summary of the preliminary results of each pilot activity, and make recommendations for the future;
- To evaluate and collect feedback from the participants of each pilot activity;

- To identify and discuss the lessons learned and best practices from each pilot activity;
- To provide recommendations and suggestions for implementing, or improving the shared facilities concept and its readiness .

1.3. Structure of the deliverable

This deliverable is structured as follows:

The Introduction chapter provides the background and context of the deliverable, the objectives and scope of the deliverable, and the structure of the deliverable.

Sections 2 and 3 and gives on overview of the six categories of shared facilities identified by WP2 and highlights their links to the pilot activities of WP3. Section 4 gives an update on the Key Performance Indicators (KPIs) reached through the described activities.

Sections 5, 6 and 7 describe the pilot activities on salinity best practice, nutrient sample storage, and carbon secondary standards, respectively, presenting their preliminary results, and providing recommendations and suggestions for updating the salinity best practice documentation, improving the nutrient sample storage techniques, and establishing a European hub for carbon secondary standards.

Section 8 summarizes and synthesizes the main findings and outcomes of the deliverable, discusses the implications and contributions of the deliverable to the EuroGO-SHIP project and the European hydrography community, and identifies the limitations and outlook of the deliverable.

2. Shared facilities overview

The six categories of shared facilities identified by WP2 are described in more detail in the following.

1. Capability: This category, evaluated for each shared facility, aims to enable data originators to access state-of-the-art analytical methods and equipment that are otherwise not available or affordable in their own countries or institutions. For example, some specialized analyses, such as transient tracers or low concentration nutrient observations, require advanced instrumentation and expertise that are not widely available or accessible. By providing access to such capability, the shared facility can enhance the scientific capabilities and productivity of seagoing research projects across Europe.
2. Certified Reference Materials (CRMs): This shared facility aims to investigate the requirement across the community for CRMs of dissolved inorganic nutrients (Nitrate, Nitrite, Silicate and Phosphate) that would be matched to the regional concentration ranges found within European seas. Having ‘certified’ nutrient reference materials is essential for ensuring the accuracy and comparability of chemical data across different regions and time periods. However, the current supply of CRMs is limited and not adequate for the European regional seas, which have diverse and variable physico-chemical characteristics. The EuroGO-SHIP project is conducting an assessment,

through a European wide survey, to determine the need and requirements for nutrient CRMs, with the exploratory phase to understand the regional demands and to evaluate the feasibility of producing nutrient CRMs in the future.

Reference Materials (RMs): There have been difficulties in recent years in obtaining crucial RMs for the carbon system parameters from the American source. It is therefore important that Europe undertakes to produce its own source of RM's, and within the EuroGO-SHIP project it is actively engaged in the development of RMs for carbon system variables. By providing this material, this shared facility can improve the quality and reliability of chemical data and services.

3. Training: This category aims to offer training opportunities for teaching and applying best practices in ship-based ocean observation, such as sampling and analysis techniques, calibration procedures, data processing, and quality control recommendations. Training is important for enhancing the skills and knowledge of both current and future members of the hydrographic community across Europe, as well as for ensuring the consistency and interoperability of hydrographic data and services. The training will potentially be delivered through various formats, such as online courses, in-person workshops, webinars, one-to-one training, and at-sea mentoring. The training will cover various topics and themes from measurements (e.g. salinity, inorganic nutrients, inorganic carbon variables, dissolved oxygen), to data management, etc.
4. Software: This category aims to develop and provide open- source software tools for data originators to perform primary quality control (QC) of their observational data on board the research ship. Primary QC is the process of checking and flagging the data for any errors or anomalies, such as instrument malfunctions, calibration errors, sampling errors, or environmental conditions. It may also include the merging and comparison of discrete sample reference data with continuous data streams and application of sample-based calibration corrections. Primary QC is essential for ensuring the quality and usability of the data, as well as for facilitating the data submission and integration, whether via automatic (e.g. submission of CTD data to the GTS) or PI-driven routes (e.g. submission to national data centres and/or international repositories). The software tools will be user-friendly, robust, and compatible with various data formats and platforms and served in open repositories via the RI website.
5. Best practices: This category aims to establish, disseminate and maintain common guidelines and protocols for all aspects of ocean observation, from planning and conducting cruises, to collecting and handling samples, to processing and submitting data. Best practices is based on the latest scientific knowledge and technological advances and is regularly updated and reviewed by experts within the consortium but in a dialogue with the wider, international community if needed. Best practice will ensure the best possible quality of data, relative to cost and time efficiency of operations. Best Practices will enable determining uncertainty estimates by consortia-agreed methods, which in turn allow to transform observational data from heterogeneous sources into a more usable product with an overall uncertainty estimation. In this way the impact of observations for a much wider community than

just hydrographic observers, can be ensured, advocating for more measurements to be recognised as Essential Ocean/Climate Variables (EOVs/ECVs).

6. A European Marine Laboratory and Equipment Pool: This category aims to investigate the possibilities for enabling European scientists to borrow or rent equipment that they need for their cruises, such as sensors, instruments, or containerised laboratories. The equipment pool will also facilitate the maintenance, calibration, and certification of the equipment prior to use. The equipment pool will be important for enhancing the accessibility and affordability of ocean observation, as well as for reducing costs and duplication of efforts across Europe.

These shared facilities are expected to provide various benefits and impacts for the EuroGO-SHIP project and the European hydrography community as a whole. They will improve the quality, reliability, and interoperability of hydrographic data and services, by ensuring the accuracy, consistency, and comparability of the data across different regions and time periods, and by facilitating and simplifying data submission and integration.

By reducing the costs and duplication of efforts, and by increasing the accessibility and affordability of the resources and capabilities needed for the observations, they will enhance the efficiency and productivity of hydrographic observations. Also, they are expected to foster the collaboration, innovation, and standardization among the hydrographic community, by providing opportunities for sharing and learning from each other, and by establishing common guidelines and protocols for ocean observation.

3. Shared facilities links to pilot activities

In this section, we present the links between the shared facilities development activities and the pilot activities that have been carried out within WP3. Scoping and defining the shared facilities concept and its categories are done within WP2, by identifying the needs and requirements of the network of hydrographic observers, and by defining the objectives, functions, and expected outcomes of each shared facility category, as well as the potential supply and demand models for their provision and access. An important activity is testing and demonstrating the shared facilities concept and its categories, by designing and executing pilot activities that are relevant to three specific shared facility categories, namely salinity best practice, nutrient sample storage, and carbon secondary standards, and by then providing feedback and recommendations for improving and expanding the shared facilities concept and its implementation.

The pilot activities are the activities that aim to test and demonstrate the shared facilities concept and its categories in practice, by conducting field experiments, laboratory experiments, and data analysis on specific topics and themes related to hydrography.

The links between the shared facilities development activities and the pilot activities are important for understanding and evaluating the progress and outcomes of the shared facilities concept and its implementation, as well as for identifying and addressing the challenges and opportunities for further development and improvement. The links also show the relevance and usefulness of the shared facilities concept and its categories for the network of hydrographic observers, as well as the benefits and impacts of the shared facilities

concept and its categories for the EuroGO-SHIP project and the European hydrography community.
The table below summarizes the links between the shared facilities development activities and the pilot activities, by indicating the type and level of interaction between each shared facility category and each pilot activity.
The type of interaction is classified as:

(i) Direct: the pilot activity directly used or contributed to a shared facility
(ii) Indirect: the pilot activity indirectly used or benefited from a shared facility
(iii) None: the pilot activity had no interaction with a shared facility category

The level of interaction is indicated by a qualitative scale (low, medium, or high).

Shared facility category	Salinity best practice	Nutrient sample storage	Carbon secondary standards
Capability	Indirect (low): The pilot activity used existing salinometers and labs, but could benefit from more advanced methods and a broader selection of equipment in the future.	Indirect (medium): The pilot activity used existing nutrient analyzers and labs, but could benefit from more specialized methods and equipment in the future.	Direct (high): The pilot activity highlights the benefit of using a laboratory that can provide stable batches of carbon RM.
Certified reference materials	Direct (high): The pilot activity used certified reference materials for salinity measurements (IAPSO Standard Sea Water), although this is a commercial product, and thus not a future shared facility of the RI.	Direct (high): The pilot activity used certified nutrient reference materials (Kanso Ltd, Japan) during the regular analysis of the stored samples thus enabling a direct comparison and quality control check for every analysis series.	Direct (high): The pilot activity will produce and distribute a batch of reference seawater for carbon system variables (alkalinity and dissolved inorganic carbon). The CO_2 CRMs (Dickson lab, SIO, US) are instrumental to check the reliability of the secondary standards
Training	Direct (medium): The pilot activity involved sharing best practices and standard operating procedures across groups, as well as providing training opportunities for some partners. Sharing procedures during training led to learning about places to improve existing practices, highlighting the benefit of direct training involving multiple experts.	Indirect (medium): The pilot activity did not involve any formal training, but it will provide advice and training on nutrient sample storage techniques in the future.	Indirect (low): The pilot activity did not involve any formal training, but the procedures for preparing stable batches of carbon RM will be documented and shared with the community.

Software	Indirect (low): The pilot activity did not use the software tool, but could have benefitted from it for performing primary quality control of salinity data on board the ship. It also could feed into modifications to the software tool based on results from pilot activity data.	Indirect (low): The pilot activity did not use the software tool, but could benefit from it for performing primary quality control of nutrient data.	None: The pilot activity did not use or benefit from the software tool
Best practice	Direct (high): The pilot activity directly used and contributed to the best practice shared facility category, by evaluating the salinity best practice documentation, revealing and discussing differences in actual practices, and planning to update it.	Direct (high): The pilot activity indirectly used and benefited from the best practice shared facility category, by following and comparing two separate methods of nutrient sample storage.	Direct(medium): The pilot activity will draft a document describing the procedure for producing a batch of RM and assigning values.
European Marine Equipment Pool	Direct (low): The pilot activity included the loan of a salinometer from one lab to another, and the procurement of the required sampling supplies and consumables for one lab by another; it demonstrated both the feasibility and some of the obstacles/requirements associated with equipment sharing.	Indirect (low): The pilot activity used existing nutrient analyzers and labs, but could benefit from more accessibility and availability of equipment in the future.	Indirect (low): The pilot activity used existing methods and equipment for producing and distributing reference seawater. However, the activity would benefit from access to an equipment pool in the future.

Table 1: Summary of the shared facilities development activities and their links to the pilot activities.

The three pilot activities are relevant to the shared facilities identified by WP2 as described in the following:

- Salinity best practice: This pilot activity is relevant to the **best practice shared facility**, which aims to establish, disseminate and maintain common guidelines and protocols for all aspects of ocean observation. By evaluating the effect of different salinometers and lab conditions on bottle salinity measurements, this pilot activity can provide feedback and recommendations for updating the salinity best practice documentation, currently based on the International GO-SHIP Manual (Kawano, 2010). It is also relevant for the **training shared facility** and **equipment shared facility**: training in salinity sampling and analysis provided to two GeoEcoMar researchers in a collaboration between NOC and IEO-CSIC highlighted both benefits of shared training and equipment, and challenges to be addressed.

- Nutrient sample storage: This pilot activity is relevant to the **best practice shared facility**, since by comparing the long-term methods of nutrient sample storage, such as freezing and pasteurization, this pilot activity can recommend the best practice for sample storage, which can ensure the accuracy and comparability of nutrient data across different regions and time periods. The methods and techniques used by the nutrient pilot activity were from the nutrient best practice based on the recently published international nutrient GO-SHIP manual (Becker et al., 2020).
- Carbon secondary standards: This pilot activity is relevant for the **certified reference materials shared facility**, which aims to provide RM for parameters of the marine carbon system, namely Alkalinity (AT) and Dissolved Inorganic Carbon (DIC). The aim is to produce RM that is suitable for the regional carbon and salinity ranges within European seas. By producing and distributing a batch of reference seawater for carbon system variables, this pilot activity can demonstrate the feasibility and benefits of creating a European hub for carbon secondary standards. This can reduce the dependency on the sole provider of carbon primary standards (the Dickson lab in the US) and offers the opportunity to produce specific batches tailored to the biogeochemistry of the different European Seas. In addition, a European hub has the potential to provide RM in alternative container with reduced stability (with respect to time) due to short shipping times.

The shared facilities development activities encountered some challenges and limitations, but also discovered some opportunities and possibilities for further development and improvement. One of the challenges is the diversity and variability of the European seas, as shared facilities will require a wide range of resources and capabilities to cover the different regions and conditions, and to ensure the accuracy and comparability of the data across different scales and domains. However, the diversity and variability of the European seas also creates an opportunity for the shared facilities development activities, as they will offer a rich and unique source of data and information for advancing the scientific and societal knowledge and understanding of the ocean, and for addressing the global and regional challenges and opportunities related to the ocean.

4. KPI Approach

This section provides an overview of how the activities and initiatives outlined in Task 3.2 align with the relevant Key Performance Indicators (KPIs) of the EuroGO-SHIP project. Progress against KPIs is regularly assessed and reported, ensuring transparency and accountability within the EuroGO-SHIP project.

KPI-2.1 determined the requirement, cost and possible supply models for new user facilities including: a) Specialist analytical capabilities to measure key parameters, such as transient tracers and low concentration nutrient analyses; b) Certified reference materials (CRMs) for nutrients and Reference Materials (RMs) for inorganic carbon; c) Training in at-sea operations, data processing and interpretation; d) Primary quality control software development and *distribution; e) Development and maintenance of shared best practices; f) A European Marine Equipment Pool (EMEP) that shares scientific equipment across Europe*

During the pilot activities in year 1, we conducted several measurements of key parameters using specialist analytical capabilities. We followed the best practices and protocols recommended by the International GO-SHIP. We also explored the possibility of creating a European Marine Equipment Pool (EMEP) that can share scientific equipment across Europe. An example of this took place in Romania, where scientists could access, and be trained on, a salinometer on loan from IEO-CSIC to be used during their cruise on R/V Mare Nigrum in the Black Sea. Further activities in this regard, currently being advanced in WP2, task 1, would be to identify the existing equipment and specialist operator skills available in different countries and institutions, as well as the gaps and needs for future cruises; to estimate the cost and benefits of establishing an EMEP, and to propose some possible supply models for its operation.

KPI-3.3 Enabled European nations to submit fully compliant cruises to International GO-SHIP supporting the International GO-SHIP mission to monitor, map and understanding oceanic heat and carbon uptake.

Our goal during the pilot cruises was to adhere to the standards and guidelines of the International GO-SHIP program as much as possible. The collaborative salinity pilot training activity had the unanticipated benefit of identifying places where common practices had diverged from GO-SHIP standards, and could be realigned at low cost. However, we also encountered some situations where the ideal conditions for applying the best practices were not met. For those cases, we will document the alternative solutions that can be adopted to compile a list of options and trade-offs (i.e., “best practices in the real world”) that can guide scientists in making decisions when facing similar challenges.

5. Pilot activity on salinity best practice

5.1. Description of the pilot activity and its rationale

Salinity is one of the key variables that characterizes the physical and chemical properties of seawater. Accurate and consistent measurements of salinity are essential for understanding the ocean's role in climate, ecosystems, and human activities. Salinity measured by CTD (conductivity-temperature-depth) sensors, the standard, is subject to (temperature- and pressure-dependent) drifts away from factory calibration settings and requires laboratory analysis of water samples collected by Niskin bottles attached to the same frame as the CTD for calibration to the highest accuracy; however, these measurements themselves are subject to various sources of uncertainty.

The laboratory analysis of salinity uses salinometers, instruments that measure the electrical conductivity of water. The resulting discrete sample salinities contain error introduced in the samples (contamination on collection, or degradation in storage) or their analysis (salinometer calibration, standardisation and linearity of response as well as uncertainties in applying the temperature- and composition-dependent conversion from conductivity to salinity); the real variability between the sample volume and the coincident-in-time CTD measurement also contributes to uncertainty in the comparison. The GO-SHIP best practice for salinity analysis was developed for a specific case (open-ocean repeat hydrography where the highest accuracy is required). To make the most of developments in instrumentation, as well as of salinity data from regions including marginal and shelf seas, and from projects with different requirements for quality and different facilities or resources available to analyse samples at sea, there is a need to update best practices for salinity sampling and analysis to reflect the effect of different methods and equipment on the resulting data quality.

The pilot activity took advantage of existing or planned cruises in European seas, and aimed to:

1) Outline the differences in salinity sample analysis procedures across several of the EuroGO-SHIP partners, with markers of where there is a consensus – a first step toward determining the range of different practices currently used across the hydrographic community, which aspects are broadly recommended, and where there is room for improvement
2) Evaluate the effects on data quality of a subset of the potential methodological variations (some expected in advance, and some revealed by objective 1) by comparing replicate samples from cruises during EuroGO-SHIP – a first step toward designing the full analyses required to determine which variations in practice are acceptable to produce different levels of data quality
3) Test the effectiveness and reveal both potential obstacles for and benefits of collaboratively sharing and updating/standardising best practices, and providing transnational access to training and analysis capabilities that would enable more groups to apply them – a first step toward developing a future shared facility

5.2. Methodology and data sources of the pilot activity

For objective 1, information on salinity sample collection and analysis procedures was requested from EuroGO-SHIP partners. A combination of standard operating procedures, salinometer manuals, and cruise or data reports were provided and their content was organised into a “default” procedure and permutations of this default, using the GO-SHIP manual (Kawano, 2010) to provide the initial outline, and analysing the procedures supplied by each partner for differences in detail as well as for aspects not addressed by the GO-SHIP best practice. Some practical variations not obvious from written procedures but revealed by discussions between partners or by the side-by-side training exercise for objective 3 were also incorporated. More details are given in the Deliverable 3.3 report.

For objective 2, the initial aim was to collect replicate samples and analyse them side-by-side on different salinometers on a dedicated cruise involving several EuroGO-SHIP partners. Due to the difficulty of scheduling such a cruise and equipment availability, an alternative involving collecting samples from multiple cruises and shipping them to different labs was explored. Replicate water samples were collected during five cruises in different regions of the European seas and North Atlantic, using two different types of glass sampling bottles. They were analysed, after varying storage times, in several ship-based or shore-side labs, each with different levels of temperature and humidity control, using two different salinometer models. The target independent variables were salinometer model, sample bottle closure type, and a well-controlled calibration standard laboratory vs different container or ship-integrated laboratories. Further details are given in the report for Deliverable 3.3.

For objective 3, there were several opportunities to pilot different aspects of sharing knowledge, skills and equipment.

- A dedicated activity was planned around GeoEcoMar cruise MN244 in July 2023, with NOC and IEO collaborating to provide both facilities (a salinometer for use onboard, salinity sampling and analysis supplies, and access to replicate analysis facilities) and in-person training for two GeoEcoMar personnel, to build GeoEcoMar's capacity to obtain sample data for calibrating CTD salinity.
- The pre-MN244 training exercise also facilitated opportunistic knowledge exchange between groups that already measure bottle salinity, revealing several areas of difference; in addition to the MN244 data, an IEO cruise in the North Atlantic in August 2023 provided some information on the effect of one of the highlighted differences.
- Preparing for replicate sample collection and comparison, where some consumables were provided by NOC to MI, and samples shipped between CNR, MI, and NOC, exercised some of the issues of customs and scheduling associated with transnational facility sharing. Salinity sampling and analysis procedures were shared between CNR and NOC online using a collaborative document prior to the April 2023 CNR cruise in the Mediterranean, serving as a trial of remote knowledge sharing.

5.3. Results and analysis of the pilot activity

Results on evaluating and updating best practices (objectives 1 and 2) are described in the report for Deliverable 3.3.

5.3.1. How much was data quality improved by providing training and equipment for salinity analysis on GeoEcoMar ship R/V Mare Nigrum for cruise MN244?

Following training and using sample bottles procured by EuroGO-SHIP, salinity samples were collected from 76 Niskins, with 50 replicates, on MN244, at depths ranging from the surface to 110 m. The 76 samples were analysed on board during the cruise between 13 and 16 July 2023 using the Autosal salinometer provided by IEO and OSIL linearity standards (for 10 psu and 30 psu) provided by EuroGO-SHIP, and following the NOC procedure (D3.3) for measuring the 4th, 5th, and 6th fills of the cell from each bottle. A few issues with analysed values, such as agreement between successive fills, were identified and flagged, resulting in 69 good samples from depths up to 90 m. The 50 replicate samples were shipped to the NOC Calibration Laboratory following the cruise and analysed on 8 September 2023 using a Portasal.

Samples analysed in the temperature- and humidity-controlled Calibration Laboratory had a constant offset of 0.00001 applied to the conductivity ratio based on 4 bottles of OSIL 10L21 and 4 bottles of P167 standard seawater (at the start and end of each set of 25 samples). Standardisation of the Autosal in operation on board the ship was tracked by running 10L21 and later 30L21; values were steady from the 13th through the morning of the 16th, but then increased through the day (not shown).

Figure 1 (left panel) compares bottle-CTD salinity residuals a) from shipboard analysis with a constant offset (-4.1×10^{-4}) to the conductivity ratio, b) shipboard analysis with a varying offset increasing to -5.1×10^{-4} and -5.5×10^{-4}, and c) calibration laboratory analysis. The depth-dependence and scatter of these residuals is not significantly different across the three versions of bottle data. Given the limited depth range, a constant based on samples between 30 and 90 m (where the background salinity gradient is relatively low, Figure 1 right panel) was used for CTD calibration. The median residual over this range is -0.0398 psu from shipboard analysis with a constant standardisation, -0.0422 psu with variable standardisation, and -0.0392 psu from laboratory analysis. The middle value was chosen, producing calibrated CTD salinity (Figure 1 right panel).

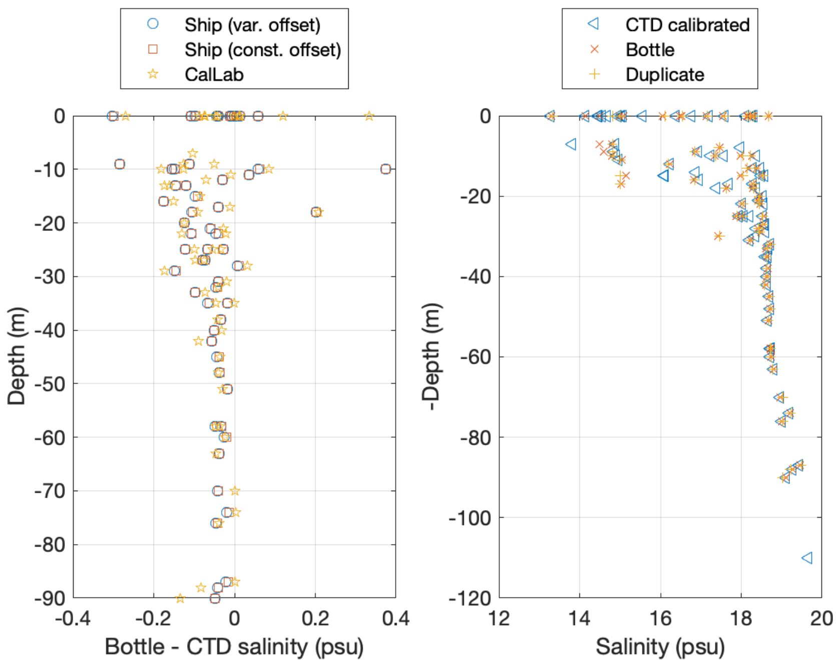

Figure 1: Residuals between bottle and CTD salinity (left), and depth profiles of bottle and CTD salinity (right), with CTD calibration derived from ship-analysed samples with a constant offset.

5.3.2. What other improvements to procedure and data arose from the pilot activities?

In the process of providing training to GeoEcoMar, two significant differences between NOC and IEO sampling and analysis procedures were identified: in the process for cleaning salinity sample bottle caps, and in the measurement of multiple readings from the same or different fills of the salinometer. Implementation of the modified cleaning procedure for caps (rinsing with DIW and drying, rather than rinsing with Niskin water) on IEO cruise RADPROF2023 increased the percentage of good bottle samples (based on bottle-CTD residuals) from 48% (on RADPROF2022) to 53% (Figure 2; Álvarez, 2023; Álvarez, 2022).

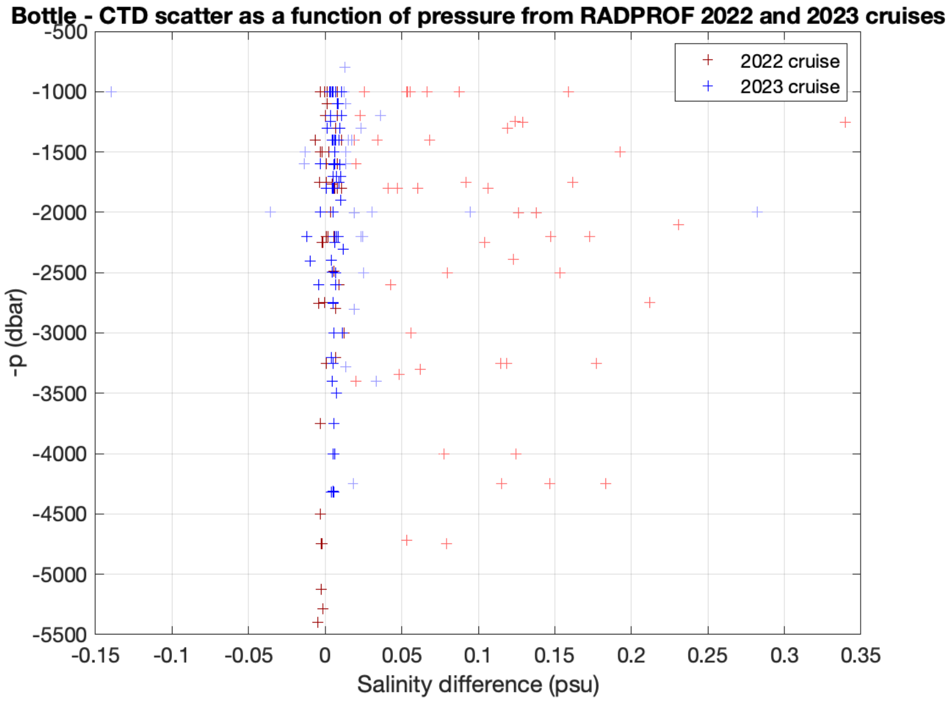

Figure 2: bottle – CTD salinity residuals before (2022) and after (2023) change to sampling technique, with bad (large residual) samples in lighter marks and good samples used for calibration in darker marks.

5.3.3. What potential obstacles to shared facilities were identified by the pilot activity?

Two major issues identified relate to shipping equipment, consumables, and samples. First, changes to the cruise schedule for MN244 meant that the salinometer sent from IEO did not return in time for the subsequent IEO cruise. Second, challenges were encountered with customs, particularly when shipping between the UK (including shipments originated by the standard seawater supplier) and EU, and where the purchaser was not the same as the recipient.

Another issue, less expected, relates to sharing methods and the best practices and training portions of a potential shared facility. While sharing sampling and analysis protocols between NOC and CNR in advance of the CNR Mediterranean cruise in April 2023 by iteration of a document by email seemed to be successful, the side-by-side operations associated with NOC and IEO providing joint training at GeoEcoMar revealed differences in methods that would quite likely not have otherwise been apparent, due to different usage of the same terminology.

Finally, the need to revise the goal for sample intercomparisons from a side-by-side evaluation on a joint cruise, to a distributed comparison involving shipping samples from multiple cruises to several different labs for analysis, due to the difficulty of cruise and equipment scheduling in the available timeframe, highlights the challenges both of quantifying the effect of procedural differences given the multiple confounding factors (D3.3 report) and equipment or even analysis facility scheduling when sharing across multiple institutions. Samples from MI cruise CE2301 were sent to NOC shortly after the cruise but could not be analysed until several months later due to issues with the available salinometers, while arrangements for shipping samples from GeoEcoMar also produced a delay of over a month.

5.4. Evaluation and recommendations

The pilot activity fostered collaboration and standardization among the hydrographic community, in addition to providing training opportunities for some partners. Through the pilot activity, EuroGO-SHIP partners identified and discussed the lessons learned not only for best practices (contained in the report for Deliverable 3.3) but also in regards to design and organisation of intercomparison exercises and planning and execution of training and facility-sharing.

Lessons learned for intercomparison exercises include:

- There is enough variation in methods, and enough implicit knowledge not necessarily written down in one document, that iteration may be required to reveal all the relevant differences.
- Side-by-side operations or training provision, as was available in the training provided before the Black Sea cruise, is a valuable way to identify differences in procedure (including significant differences, as in the IEO example above).
- A side-by-side comparison of sampling and/or analysis would reduce the degrees of freedom, but would still leave questions about factors including variance between machines of the same model, and occasional or intermittent issues with samples, salinometers, or ambient conditions.

Lessons learned for shared facility design include:

- Planning for equipment sharing as well as for shared analysis facilities, if done on a more than occasional basis, needs to account for a margin of error for schedule changes (or accept the risk to follow-on expeditions), and to involve all relevant equipment users and facility operators from the start, as well as a defined method for prioritisation of different users' requirements.
- Budgeting for equipment sharing should include costs for maintenance and servicing of the shared equipment, including consideration of the additional wear that may result from higher utilisation or more shipping.
- Shipping of consumables and samples can require considerable time dealing with customs documentation and queries; a shared facility might be able to provide expertise to streamline this work.
- Planning for sample analysis ashore needs to involve shore-side facility managers to ensure availability and prevent additional storage time.

6. Pilot activity on nutrient sample storage

6.1. Description of the pilot activity and its rationale

Dissolved inorganic nutrients are important variables that characterize the biogeochemical cycles and productivity of the ocean. Accurate and consistent measurements of nutrients are essential for observing and understanding important trends as ocean biogeochemistry responds to climate change, ecosystem trends and anthropogenic pressures. However, nutrient measurements are subject to various sources of uncertainty, such as instrumentation, calibration, sampling and storage biases, which can mask or to be superimposed on their natural variability due to environmental conditions. Therefore, there is a need for applying current best practices for nutrient sampling and analysis, which can ensure the quality and comparability of nutrient data across different regions and time periods (GO-SHIP Repeat Hydrography Nutrient Manual: The precise and accurate determination of dissolved inorganic nutrients in seawater, using continuous flow analysis methods; i.e. Becker et al 2020). Observations and results from the pilot activities can then be used for future updates of this GO-SHIP manual.

One of the key factors that affects the quality and reliability of nutrient measurements is the sample storage method. Nutrient samples are usually collected by Niskin/Go-Flo water sampling bottles mounted on a CTD Rosette frame, which is lowered through the water column and samples can be taken at the required depths. Nutrient samples are then transferred to plastic bottles and stored until analysis. The best practice and recommended process is to analyse the samples as soon as possible once the water is returned on board the research ship. However, this is often not possible due to various reasons like ship size, lack of analytical facilities or trained personnel etc. The only option then is for the storage of the samples for later analysis back in the land laboratory, and then the storage time and environmental conditions during sample storage can influence the nutrient concentrations in the samples, due to contamination and/or biological or chemical processes that can alter the nutrient speciation and concentrations. Therefore, there is a need for evaluating and comparing some of the more widely used methods of nutrient sample storage, and for recommending the best practice for sample storage accordingly.

The pilot activity on nutrient sample storage aimed to compare the long-term methods of nutrient sample storage, using two of the most widely used methods globally which are freezing and pasteurization, and to provide feedback and recommendations to the community and for then updating the nutrient best practice documentation. Freezing has always been a preservation method used for many years by laboratories because it is a simple and effective method of inhibiting biological activity and preventing nutrient sample alteration, and does not involve costly equipment nor chemicals, however, there is often issues such as bottle breakage and leakage and ensuring the samples are returned to the land laboratory still frozen, which depends on couriers. Often samples are lost, especially if shipping back over long distances from the port where the research cruise ends. Pasteurization (Daniel et al. 2012) is a method of heating the samples to a high temperature, which can kill any microorganisms and stabilize the nutrient concentrations. As a storage technique it has been much more limited in its use as scientists have favoured freezing as it is seen as generally reliable, furthermore pasteurization involves initial extra costs with the purchase of a suitable oven, however, once pasteurized the samples can then be handled and shipped at room temperature and it avoids any problems with defrosting etc.

The pilot activity involved three partners from the EuroGO-SHIP consortium. The pilot activity took advantage of existing or planned cruises in different regions of the European seas, such as the North Atlantic and the Mediterranean Sea. The pilot activity also involved sharing best practices and standard operating procedures across groups, as well as providing possibilities of training opportunities between partners.

6.2. Methodology and data sources of the pilot activity

The pilot activity used a combination of field experiments, laboratory experiments, and data analysis to compare the long-term methods of nutrient sample storage. The main steps of the pilot activity were:

- Field experiments: Collecting replicate water samples for long-term nutrient analysis during two cruises in different regions of the European seas, using different types of sampling bottles (60ml and 125-mL, HDPE bottles and 30-mL, borosilicate glass bottles with teflon screw cap for ammonium) and different types of filters (0.2-µm Mixed Celluloses Esters (MCE) membrane filters and 0.2-µm PTFE filters). The cruises were:
  - PML/NOC cruise at the Porcupine Abyssal Plain in the NE Atlantic in May 2023.
  - CNR cruise in the Gulf of Trieste in May 2023.
- Laboratory experiments: The water samples taken during the research cruises were stored using either freezing or pasteurization and then analyzed over the course of 12-15 months in 2 different laboratories. The analysis method used was segmented flow colorimetric analysis for all the nutrients. The determination of ultra-low concentrations of orthophosphate in the seawater samples in the Gulf of Trieste (< 0.05 µmol P L^{-1}) was performed by a high-precision manual colorimetric method. The labs were:
  - PML laboratory in Plymouth, UK
  - CNR laboratory, Trieste, Italy
- Data analysis: The environmental conditions at the time of the samplings were investigated in these different marine systems, to highlight the significance of the nutrient samples. All sub-samples that were needed to assess treatment and storage methods were collected in triplicate and nutrient concentrations were expressed as mean and standard deviation. Possible temporal trends of nutrient concentrations and/or the alteration of the precision of nutrient determinations will be assessed by statistical analyses.

6.3. Results and analysis of the pilot activity

The preliminary results for the water column sampling sample storage for the pilot activity showed that:

- For CNR Cruise in the Gulf of Trieste (May 2023), nutrient samples were collected at the surface and 1 m above the sea floor in six sampling stations. Meteorological observations and CTD casts indicated the occurrence of instable weather conditions and the presence in the gulf of high-salinity and low-nutrient coastal waters. These characteristics are typical of periods with a strong circulation and mixing and a scarce river runoff.

- PML carried out the at-sea nutrient analysis using a 5-channel SEAL analytical segmented flow colorimetric autoanalyzer to analyse Nitrate, Nitrite, Silicate, Phosphate and Ammonium, during the National Oceanography Centre (NOC) Porcupine Abyssal Plain cruise on the RRS James Cook (JC247) cruise in May 2023. The first water sampling was carried out at 50m depth just offshore from Plymouth, South-West England at 50° 15.00' N, 4° 13.02' W. This was also the site of the PML long-term survey station L4, which is part of the Western Channel Observatory. Further samples were taken at the Whittard Canyon and the Porcupine Abyssal Plain stations at depths of 200m, 500m and a deep sample with high nutrients at 4824m. These then covering a wide range of nutrient concentrations that would be typically found in the seas across Europe. At each of the 4 sampling depths 45 HDPE bottles were taken for both Freezing and Pasteurization, plus 45 Glass bottles just for Pasteurization and ammonium analysis were taken and stored appropriately for the future analysis. Importantly, in order to ensure comparability between all the sample analytical results Certified Nutrient Reference Materials (CRMs) were analysed alongside the at-sea and all the regular laboratory analyses, which will allow direct comparisons of all the results relating back to the CRM results and so will give good analytical certainty when comparing the results and long-term trends. The CRM's were sourced from KANSO Ltd, Japan, and were lots CI and CH, the original source of which originated from the North Atlantic Ocean.
- The effectiveness of freezing and pasteurization as methods of storage of filtered and unfiltered nutrient samples is still under evaluation through periodic laboratory analysis until June 2024.

6.4. Evaluation, best practices and recommendations

The pilot activity identified and discussed the lessons learned and best practices from the field experiments, laboratory experiments, and data analysis. Some of the preliminary lessons learned and best practices were:

- The choice of the storage method should be based on the availability and affordability of personnel and equipment, and the appropriate space and facilities available on research vessels and boats, the distance, difficulties and risks of shipping of samples, as well as on the expected storage times and frequency of laboratory analyses.
- Sample treatment and storage methods should follow standard operating procedures that, however, should be chosen based on the expected physico-chemical characteristics of the samples: e.g. salinity levels, turbidity, plankton biomass, nutrient concentration levels. These characteristics can strongly vary from oligotrophic to eutrophic marine ecosystems.
- Sample treatments and storage methods can have distinct effects on individual nutrients (nitrate, nitrite, ammonium, phosphate and silicate). For this reason, distinct

analytical methods might be applied in case only some of these nutrients have to be analysed.

The completion of the analysis of the results of this pilot activity will provide more detailed recommendations and suggestions for updating the nutrient best practice documentation, which is currently based on the International GO-SHIP Manual (Becker at al., 2020). It will be also useful to revise and update the guidelines and protocols for nutrient sampling and analysis, considering the effect of different methods of sampling and storage of nutrient samples, and providing practical tips and examples for minimizing negative effects.

7. Pilot activity on carbon secondary standards

7.1. Description of the pilot activity and its rationale

The global marine inorganic carbon community depends on measurements of discrete water samples for total dissolved inorganic carbon (DIC), total alkalinity (TA) and pH to characterize the inorganic carbon content of seawater. The aim of this measurements reaches from ocean acidification experiments to observations of global trends of anthropologic carbon in the deep sea. The quality of these kind of measurements increased immensely since the introduction of standard seawater that is certified for its DIC and TA content in the 1990s through the laboratory of Andrew Dickson in the US (e.g. Dickson, 1993). The provision of such certified reference materials (CRMs) is uncertain in the future, which is unsettling the global oceanographic community. In addition, the supply of primary CRMs from the Dickson lab is limited and costly, and there is a need for alternative sources of primary or at least secondary CRMs, especially in Europe. Therefore, efforts have been undertaken by the International Ocean Carbon Coordination Project (IOCCP) and the Global Ocean Acidification – Observation Network (GOA-ON) to put the provision of CRMs for the measurements on a broader international basis, including regional hubs, one of which is supposed to be in the European/African region. The aim of this pilot activity was to produce and distribute a batch of secondary carbon reference material (RM) for the European marine carbon community, following the procedures developed at the Dickson lab. Secondary RMs are needed to ensure the accuracy and comparability of DIC and TA measurements across different regions and time periods, as well as to calibrate and validate sensors and instruments. This pilot activity was intended to demonstrate the feasibility and benefits of preparing and distributing secondary RMs at GEOMAR, and to contribute to the global effort for establishing best practices for this process.

7.2. Methodology and data sources of the pilot activity

The pilot activity involved the following steps:

1) Preparation of a batch of seawater with North Atlantic Ocean water at GEOMAR:

Following the procedures developed at the Dickson lab to minimize contamination during production. The seawater was poisoned with mercuric chloride and stored in a dark and temperature-controlled room. At least three days before the water was filled into glass

bottles, a submersible pump was inserted to ensure turbulent mixing within the batch and to ensure homogenization. To provide a constant atmosphere above the seawater, either a gas bottle with a constant CO_2 content was connected or a pump providing clean atmospheric air at a flowrate of approximately 50 mL/min (see Fig. 3). We also connected a UV sterilization unit, which is not shown in the figure.
In addition to the pump a thermosalinograph (Seabird SBE37) was deployed in the batch to also estimate salinity. This can also be done with discrete samples, if possible (Note: the water is poisoned!).

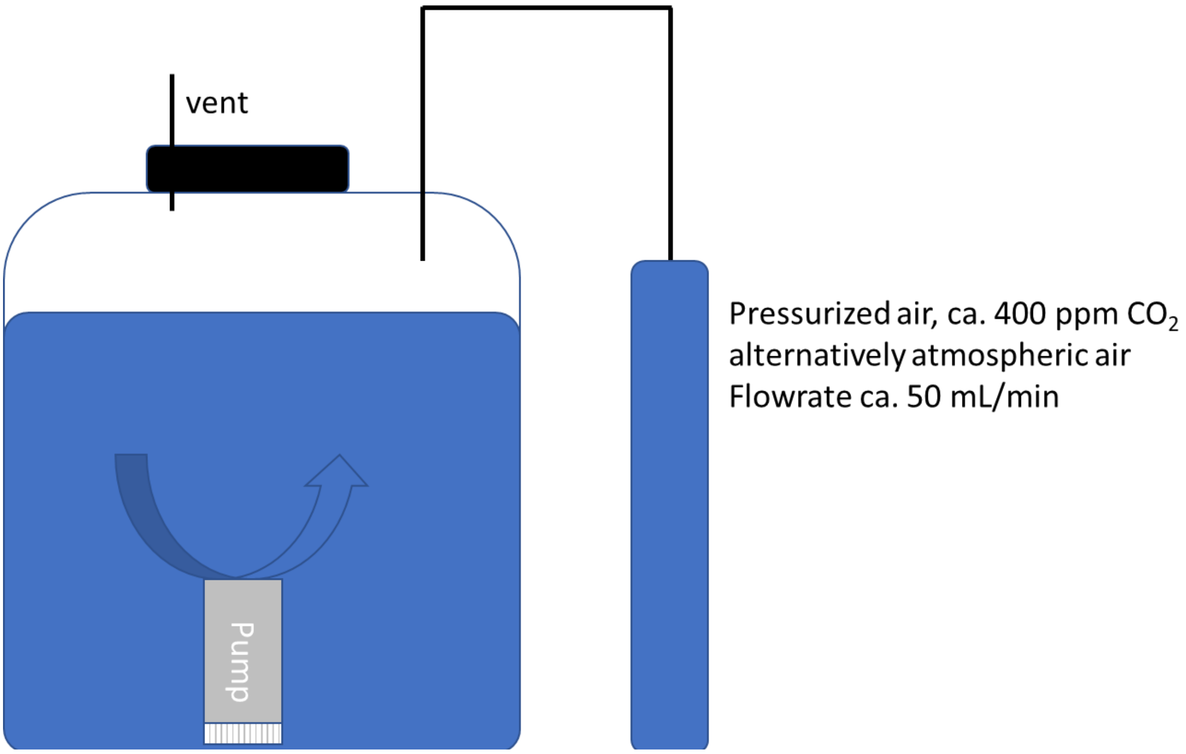

Figure 3. Schematic of the batch preparation for carbon secondary reference material.

2) Bottling of the seawater:

The pump in step 1 is turned off before the sampling procedure and the flowrate of the headspace gas is increased to ca. 100 mL/min. This is to ensure that the headspace is renewed faster than room air can enter even when removing water for the samples. Fig. 4 shows a schematic of the setup used to fill the batch into glass bottles. We used borosilicate glass bottles (250 mL and 500 mL) that were rinsed and tempered at 500°C for approximately 5 hours. A hose was introduced to the bottom of the tank, leading to a peristaltic pump, that was connected to a foot pedal to turn the pump on/off.

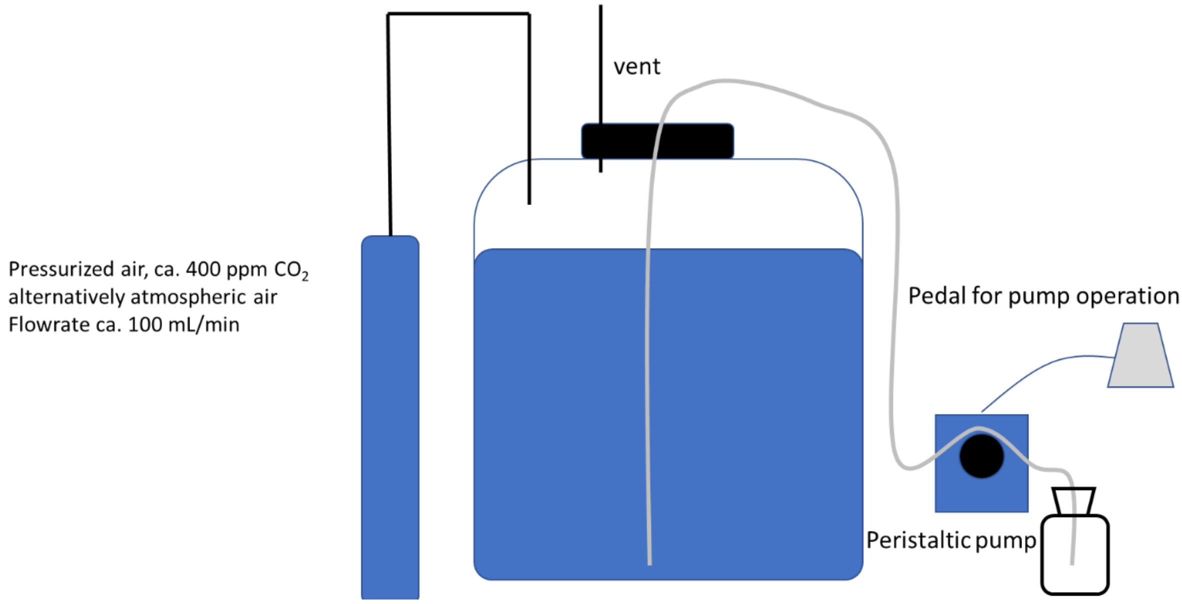

Figure 4. Schematic of the sampling setup for filling the batch into bottles.

The free end of the hose was then introduced into a clean glass bottle and the bottle was filled from the bottom to the top, leaving some headspace and immediately closed as recommended in Dickson et al (2007).
We will also fill some water in air-tight plastic bags (volume: 5L) and test their suitability for short term stability (up to two months) of DIC and TA.

3) Evaluating the batch:

The filled bottles are kept in a dark place at room temperature for a few days. Then at least eight samples are analyzed for DIC and TA. We use a SOMMA system with coulometer for DIC and a VINDTA system for TA determination. The values of these eight bottles should agree within 2 µmol/kg (given the instruments are running well), otherwise something must have gone wrong with the sampling and the batch should not be used. The instruments will be calibrated using carbonate standards and the results will be quality-controlled using CRMs from the Dickson laboratory.

4) Distribution of sub-samples of the bottled seawater to the Dickson laboratory

If feasible sub-samples of the batch will be sent to the Dickson laboratory in the US for independent evaluation. The Dickson laboratory is under re-organization at the moment, and it is not clear if this task will be completed. In this case the samples will be distributed to other labs within Europe for independent evaluation.

7.3. Results and analysis of the pilot activity

GEOMAR moved its laboratory into a new building. The process of moving and setting up the laboratory in the new building at the time of writing time (April 2024) is not completed. Not all instruments are working properly, yet. The first batch will be bottled by the end of April and can be distributed to project partners by mid-May 2024.

7.4. Evaluation, best practices and recommendations

The group at GEOMAR already prepared two batches of secondary reference material but improved certain steps for the application in the EuroGO-SHIP project. The pilot activity derived some lessons learned and best practices from the pilot activity, such as:

- There is a strong need for bigger sampling containers with shorter storing times to be used during research cruises. First tests have shown that this is achievable for TA with an uncertainty of ±3 µmol/kg, but not for DIC.
- It could be shown that using the method described above a stable batch of seawater reference material can be prepared. One prerequisite is the availability of clean seawater. Planned cruises should be used to sample big quantities (1 m^3) of seawater with low biological content. This samples should be stored and observed for at least a year to ensure that there is no significant biological activity.

8. Conclusions

This deliverable reports on the pilot activities relevant to shared facilities that were carried out in the framework of task 3.2 of the EuroGO-SHIP project. The pilot activities aimed to demonstrate a key function of a EuroGO-SHIP RI, that is revising best practice documentation when it becomes necessary. Three pilot activities were undertaken, to inform best practices for salinity analysis, nutrient sample stability and carbon secondary standards. The deliverable describes the rationale, methodology, data sources, results, analysis, evaluation, best practices and recommendations of each pilot activity, as well as the challenges and limitations encountered, and the lessons learned from the experience. The pilot activities demonstrated the ability and readiness of the EuroGO-SHIP consortium to revise best practice documentation when necessary, based on the latest scientific knowledge and technological advances. Furthermore they will enhance the capacity and capability of the EuroGO-SHIP consortium to access state-of-the-art analytical methods and equipment, as well as reference materials for carbon, that are otherwise not available or affordable in their own countries or institutions. In general, collaboration and communication among the EuroGO-SHIP partners and other stakeholders in the European and global hydrography community has gone a step forward.

The pilot activities also faced some limitations and challenges that need to be addressed and overcome for the successful implementation and sustainability of the EuroGO-SHIP RI and its shared facilities.

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