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(Article begins on next page)

# Challenges in $p\text{CO}_2$ measurement: lessons learnt from the MINKE project

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**Abstract**—The project MINKE “*Metrology for integrated marine management and knowledge transfer network*”, ended in March 2025, aimed to bring together 16 key European marine metrology research infrastructures to create an innovative ‘quality of oceanographic data’ framework, starting from the Essential Ocean Variables (EOVs) as the key parameters to monitor the oceans, and adopting a multidimensional framework of data quality. One of the EOVs that was investigated is the partial pressure of  $\text{CO}_2$ , which represents an indicator to monitor the course and potential ecological impact of ocean acidification on the biological processes in the oceans. In the present paper, we summarise the key issues and the potential actions to improve the quality of the measurements of  $p\text{CO}_2$  in the oceans, as well as the challenges to be faced in the upcoming years.

**Keywords**—*partial pressure of  $\text{CO}_2$ , metrological challenges, calibration, uncertainty evaluation*

## I. INTRODUCTION

The project MINKE “*Metrology for integrated marine management and knowledge transfer network*”, ended in March 2025, aimed to bring together 16 key European marine metrology research infrastructures (oceanographic institutes, National Metrology Institutes (NMIs) and Designated Institutes (DIs), Universities, Fablabs) to create an innovative “quality of oceanographic data” framework, among the different European actors dealing with the monitoring of the Essential Ocean Variables (EOVs). The cooperation and networking among oceanographic institutes and NMIs/DIs represents a fundamental aspect in this context, to assure the reliability and metrological traceability of measurement results [1]. EOVs are key parameters to monitor the oceans and the marine ecosystems, adopting a multidimensional

framework of data quality based on the concepts of accuracy and completeness [2]. Data quality is the key element in ocean and coastal observing systems, to provide reliable measurements for developing evidence-based environmental policies. The MINKE project focused on all data quality aspects, with a special emphasis on those related to metrological concepts. The project also proposed a new vision in the design of marine monitoring networks, integrating the concepts of accuracy and completeness as driving components of quality in data acquisition. This idea was intended to create an optimal metrological framework and a combined system integrating the information provided from the selection of few reference points for regular monitoring, using top-level instrumentation to maximise the measurement accuracy, and the use of low-cost observational systems, in the attempt to cover all the potential points of measurement, with the final goal to maximise the completeness of the datasets [2].

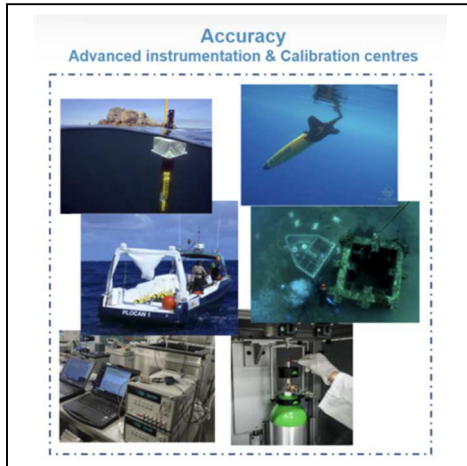


Fig. 1. Examples of research infrastructures involved in the MINKE project to assure accuracy to the measurement results [2].

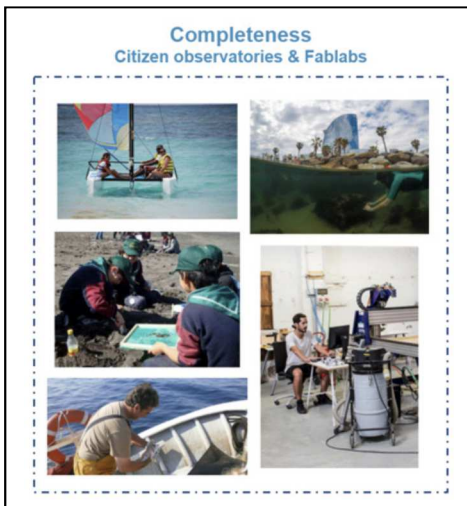


Fig. 2. Example of facilities involved in the MINKE project, to assure completeness of the data collected [2].

The several EOVs defined by the Global Climate Observing System (GCOS) [3] are useful to study the status of the marine environment and to obtain quantifiable indications to monitor the phenomena occurring in the oceans and to relate them to the changes occurring on the global scale in all the environmental compartments. EOVs can be considered key indicators for ocean life and their study is required to support policy makers and international management in the framework of climate change studies.

## II. PARTIAL PRESSURE OF CO<sub>2</sub>

The carbon system is in a delicate balance and high quality observations are required. The variables needed to constrain the carbon system at a point in space and time can be any two among the following ones: Dissolved Inorganic Carbon (DIC), Total Alkalinity (TA), partial pressure of carbon dioxide ( $p\text{CO}_2$ ) and pH on the total hydrogen ion scale ( $\text{pH}_T$ ), and associated physical variables, e.g. temperature and salinity [4]. DIC, TA,  $\text{pH}_T$ , and  $p\text{CO}_2$  describe the seawater CO<sub>2</sub> system and are chemically interrelated. Some variable pairs – including that of  $\text{pH}_T$  and  $p\text{CO}_2$  – are more problematic

due to their correlation in seawater, which magnifies the uncertainties of the other variables calculated using them.

The  $p\text{CO}_2$  is one of the few variables of the carbon system in water (freshwater or seawater) that can be directly measured *in situ*. The  $p\text{CO}_2$  in air that is in equilibrium with (a sample of) seawater is defined as the product of the amount fraction of CO<sub>2</sub>,  $x(\text{CO}_2)$ , in the equilibrated gas phase by the total pressure ( $p$ ) of equilibration [5]:

$$p\text{CO}_2 = x(\text{CO}_2) \cdot p \quad (1)$$

The importance of  $p\text{CO}_2$  measurement lies in its usefulness in the determination of the fugacity of CO<sub>2</sub>,  $f(\text{CO}_2)$ . This parameter is not the same as  $p\text{CO}_2$  because it involves the non-ideal nature of the gas phase of CO<sub>2</sub>. The magnitude of the fugacity coefficient (the ratio of fugacity to partial pressure) is a function of both temperature and gas phase composition [5]. In addition to its role in the air-sea gas exchange and the Earth's CO<sub>2</sub> system,  $p\text{CO}_2$  is also a helpful indicator of the course and potential ecological impacts of biological processes like photosynthesis and respiration.

## III. CHALLENGES IN $p\text{CO}_2$ MEASUREMENT

### A. Challenges in calibration

There is a wide availability of *in situ* sensors and analytical methodologies for monitoring  $p\text{CO}_2$  in marine environments, but there are still many open issues, such as the differences in adopted calibration methodologies, the application of non-validated procedures and the lack of suitable metrological traceability paths and of operational harmonisation for field measurements. Another problem is related to the scarcity and expensiveness of proper reference materials to calibrate the instruments used for  $p\text{CO}_2$  monitoring.

At present, the main techniques employed by commercially available sensors used for measuring  $p\text{CO}_2$  in seawater are based either on the equilibration of a carrier gas phase with a seawater sample and the determination of the CO<sub>2</sub> that diffuses through by means of non-dispersive infrared (NDIR) sensors, on reagent-based colorimetry (COLM) or on species-specific solid-state detectors (e.g. optodes). This last technology is still in an early stage of development compared to the other two. Another technology recently applied is cavity ring-down spectroscopy (CRDS).

In oceanographic practice, seawater variables are typically measured according to internal standard operating procedures (SOP). These are based on state-of-the-art best practice documents, prepared and/or approved by most relevant oceanographic institutions, such as the IOCCP Report No. 8 “Guide to Best Practices for Ocean CO<sub>2</sub> Measurements” [5], many of which are openly available in internationally recognised repositories of the oceanographic community like the IOC-UNESCO Ocean Best Practice System (OBPS). For  $p\text{CO}_2$  in particular, two SOPs [5] should be mentioned:

- SOP 4: Determination of  $p\text{CO}_2$  in air that is in equilibrium with a discrete sample of seawater;
- SOP 5: Determination of  $p\text{CO}_2$  in air that is in equilibrium with a continuous stream of seawater.

The Ocean Observatories Initiative (OOI) best practices [6] include a section on CO<sub>2</sub> sensors and their deployment on moorings. This document also contains guidance on common

Data Quality Issues (section 4.4), with a particular focus on issues common to carbonate chemistry sensors, and more in general, recommendations for End-User Data Processing (section 4.5). In the case of SOCAT, the Surface Ocean CO<sub>2</sub> Atlas [7, 8], for an accuracy estimate of better than 2  $\mu\text{atm}$  (A or B level), seven standard operating procedures (SOP) must all be fulfilled and properly documented. Generally, sensors need sufficient metadata to assess their overall accuracies in practical applications. The IOCCP [9] provide some information on how commonly used sensors have performed in field and laboratory comparison studies.

Table 1 reports a list of typical sensors commonly used for  $p\text{CO}_2$  determination in seawater.

TABLE I. A SELECTION OF COMMONLY USED MARINE  $p\text{CO}_2$  SENSORS

Manufacturer / model / principle	Precision	Range	Accuracy	Pressure rating
Contros Hydro-CO <sub>2</sub> (NDIR)	<1 $\mu\text{atm}$ (resolution)	200-1000 $\mu\text{atm}$	5 % of reading (initial)	6000 m
Pro-Oceanus CO <sub>2</sub> -Pro (NDIR)	0.01 ppm	0-2000 ppm (max)	0.5 %	6000 m (max)
Picarro G2401 (CRDS)	<0.05 $\mu\text{atm}$	300-500 $\mu\text{atm}$	0.5 $\mu\text{atm}$	Surface only
Sunburst SAMI-CO <sub>2</sub> (COLM)	1 $\mu\text{atm}$	150-700 $\mu\text{atm}$	3 $\mu\text{atm}$	600 m (max)
Aanderaa Optode (OPT)	2 $\mu\text{atm}$ , between 200-1000 $\mu\text{atm}$	0-50000 $\mu\text{atm}$	2-75 $\mu\text{atm}$	6000 m
MAPCO <sub>2</sub> (NDIR)	0.6 $\mu\text{atm}$ , between 100-600 $\mu\text{atm}$	0-1000 $\mu\text{atm}$	0.3 $\mu\text{atm}$	Surface only
General Oceanics 8050/8060 (NDIR OF-CEAS)	\	0-3000/0-10000 $\mu\text{atm}$	2 $\mu\text{atm}$	Surface only

Whatever the sensor used, verifying that specifications are being met when it is operating in the field remains a huge challenge, both because the specifications provided by the manufacturer do not highlight the contributions to the standard uncertainty attributable to the repeatability and reproducibility of the instrument and because these contributions themselves would have to be estimated directly in the field by the user. This latter is a difficult task due to the variability of the measurand attributable to variations in boundary conditions (seawater temperature, current, etc.) or to systematic effects such as bio-fouling (which can have a certain influence especially on instruments mounted on mooring for long time series). It is often the detector precision and accuracy that are quoted by the manufacturer, whereas the whole system including equilibration of CO<sub>2</sub> (e.g. across a membrane) is used for the actual measurements and replicates.

Where the measurement relies on an equilibration step involving the seawater that is being sampled with a gas (usually, air) or a liquid phase (usually, a reagent mix), the stability of the temperature and pressure conditions of

equilibration is of paramount importance. Knowledge of the equilibration temperature is particularly crucial because  $p\text{CO}_2$  in seawater varies strongly with temperature, and a correction in the final measurement results will be necessary to compensate for the difference between the seawater sample temperature in the equilibration step and the actual *in situ* seawater temperature, if they differ significantly. Calibrating such sensors focusing primarily on the detector does not always account for the dependencies on the equilibration stage.

### B. Challenges in uncertainty evaluation

In recent years, technologies have been evolving with a whole new generation of sensors and instruments measuring  $p\text{CO}_2$  in the ocean (both at the surface and at depth) entering the market. The evaluation of the measurement uncertainty is a fundamental step in this area of measurement to assure comparability of collected data.

To assure a long-term coherent and reliable observation of the four variables DIC, TA,  $\text{pH}_T$ , and  $p\text{CO}_2$  in the ocean, some crucial issues should be addressed. A major metrological issue is represented by the inconsistencies for the measured variables, that have been highlighted by reviews of observations from oceanographic cruises, and of uncertainties in the seawater CO<sub>2</sub> system variables. This would affect the calculations of the variables, when two out of the four variables are measured [10, 11]. These inconsistencies become important for ocean monitoring when combining data from various sources, e.g. measurements of  $\text{pH}_T$  and  $p\text{CO}_2$  from autonomous sensors with discrete determinations of DIC and TA, obtained using benchtop instrumentation. The observed inconsistencies might come from uncertainties of the physicochemical model of the seawater CO<sub>2</sub> system, e.g. if all relevant components (i.e. organic compounds) are not taken into account or if the equilibrium constants are not adequately quantified. In addition, measuring each variable is not straightforward, so inconsistencies might additionally result from methodological and/or technical issues.

Currently, there are two main objectives behind observations to monitor  $p\text{CO}_2$  activity in seawater:

- to identify relative spatial patterns and short-term variation (the “Weather” objective), where a relative measurement uncertainty of about 2.5 % (10  $\mu\text{atm}$ ) is called for;
- to assess long-term trends with a defined level of confidence (the “Climate” objective), where a more stringent relative measurement uncertainty of 0.5 % (2  $\mu\text{atm}$ ) is desired.

In practice the specifications for seawater  $p\text{CO}_2$  measurements can vary depending on the operational setting (e.g. the coast, the open ocean, etc...), the objectives of the observing activity and the inherent limitations of the employed measuring technologies. The *Integrated Carbon Observation System* (ICOS) recommended accuracy specifications for marine  $p\text{CO}_2$  measurement [12]. ICOS is a distributed pan-European research infrastructure producing high-quality data on greenhouse gas concentrations in the atmosphere and carbon fluxes between the atmosphere, the land surface and the oceans.

### C. Possible solutions

- *Calibration*

Best practices recommend validation using calibration gases to account for the equilibration step of  $p\text{CO}_2$  measuring systems, when validating  $p\text{CO}_2$  data, but this is still not widely adopted for membrane-based systems, which form the bulk of the measurements made on submerged platforms. It is also possible to validate membrane equilibrators systems against systems utilising gas standards. A significant limitation of gas-based  $p\text{CO}_2$  sensors is the difficulty of providing regular calibrations against standard gases, which makes challenging the assessment of their accuracy over extended periods, especially in stand-alone applications. Newer versions of gas-based  $p\text{CO}_2$  sensors sometimes allow on-board control of a gas port for introduction of reference gases; if a sensor is to be used for on-board applications, a version that enables external manual calibration is recommended.

For field applications, users of any chemical sensor that is not calibrated while deployed, should calibrate the sensor before and after long-term deployments to examine any potential drift. The quality and uncertainty of the calibration gases used in the original factory calibration and any subsequent recalibration are critical factors for the sensor accuracy and the uncertainty of measurement results.

- *Uncertainty evaluation*

The ICOS Ocean Thematic Centre (OTC, <https://www.icos-otc.org/>) suggested changing the ICOS criteria for  $p\text{CO}_2$  sensors for fixed ocean stations (FOS) measuring continuous or quasi-continuous samples, to  $\pm 10 \mu\text{atm}$ , which is in line with the SOCAT criteria [7,8] for alternative sensors.

Jiang *et al.* [13] suggested that, when  $p\text{CO}_2$  references are available for the correction of the measurement data, the uncertainty of the corrected sensor result is similar to, and largely determined by, the uncertainties of the references.

Another suggestion was to perform parallel  $\text{CO}_2$  measurements in the field. This means that, when a  $\text{CO}_2$  sensor is to be retrieved, the user should ensure that the old sensor and the new calibrated sensor are measuring in seawater side by side for a couple of hours before the old sensor is retrieved. This procedure can help in determining a drift of the old sensor and act as a validation of that sensor. This field exercise requires that the user has more than one  $p\text{CO}_2$  sensor available.

Finally, the collection of discrete samples over a wide range of  $p\text{CO}_2$  concentrations for the determination of other carbonate variables is recommended to provide information useful for quality assessments of a  $p\text{CO}_2$  sensor (also in the light of the existing natural variability).

In this context, intercomparisons such as those organised by the ICOS OTC can be of fundamental relevance, and have the following aims:

- to compare the performance of instruments and sensors that are used within the ICOS community over a range of temperatures and  $p\text{CO}_2$  levels;
- engage instrument suppliers and manufacturers to collaborate with the observational community to

reach a high level of standardisation in operating  $p\text{CO}_2$  sensors and instruments;

- support the ocean observation community in the choice of the appropriate sensors for their application.

### IV. CASE STUDIES

A case study from NOC (UK) compared different methods to estimate and measure  $p\text{CO}_2$  *in situ* and to validate  $p\text{CO}_2$  sensors [14]. Year-round  $p\text{CO}_2$  data is collected at NOC's Porcupine Abyssal Plain Sustained Observatory (PAP-SO, 49N 16.5W, <https://projects.noc.ac.uk/pap/>) in the Northeast Atlantic with two Pro-Oceanus  $p\text{CO}_2$  sensors, one measuring in air (IR absorbance after equilibration) and the other in water (gas tension device). The sensor is declared to be accurate to  $\pm 0.5\%$ , with a resolution of 0.01 ppm. Both the sensors perform a daily zeroing to account for drift during deployment.

On the annual turnaround cruises, there is an opportunity to run underway  $p\text{CO}_2$  measurements on-board for comparison. This is usually done using an underway SubCtech system, calibrated to 2 onboard standards. The system uses NDIR LI-COR (USA) detection with membrane equilibration. The accuracy is quoted as  $< 1.5\%$ , with a 0.01 ppm resolution for  $\text{CO}_2$ .

The Pro-Oceanus sensors deployed at the PAP-SO gave results similar to the SubCtech system run during the annual turnaround cruises. This demonstrated that onboard measurements could be used to ground truth the *in situ* sensor data of the observatory.

Both the Pro-Oceanus and SubCtech systems were under-reading by  $\sim 20\text{-}30 \mu\text{atm}$  compared to the calculated  $p\text{CO}_2$  from DIC and TA measurements. However, this parameter pair is not ideal for calculating  $p\text{CO}_2$ . ICOS recommends pH measurements are taken *in situ* alongside DIC, as this is a better parameter pair for calculations. ICOS best practices [12] prescribe taking triplicate samples for analysis of pH and one of either DIC or TA to calculate  $p\text{CO}_2$ . Samples are to be taken as near as possible to the sensors, at the start and end of the yearlong sensor deployments.

The GO (General Oceanics) system is the 'gold-standard' system, as it uses a LI-COR detector, showerhead equilibration, and is ground truthed to multiple gases. The GO system is the main instrument used on the ships of opportunity (SOOP) within ICOS OTC, and has a quoted accuracy of  $\pm 2 \mu\text{atm}$ . It was used as the reference system within the ICOS intercomparison of  $\text{CO}_2$  equipment [15]. The GO system was fitted to the ship during 2024, and will be used to make future underway and *in situ* comparisons at the PAP-SO, alongside calculations of  $p\text{CO}_2$  from samples of pH and DIC.

A second case study developed from ENEA (Italy) was a metrological in-field comparison between a Pro-Oceanus  $\text{CO}_2$ -Pro CV probe and a multi-parametric monitoring system (OceanPack-RACE, produced by SubCTech) for the measurement of temperature, salinity, oxygen and  $p\text{CO}_2$ , especially designed for racing yachts [16,17].

The study was articulated in two stages, a first step concerning on-bench activity for the metrological check of the standard probe by means of a certified  $\text{CO}_2$  gas mixture.

A Pro-Oceanus CO<sub>2</sub>-Pro CV probe was used for measuring the CO<sub>2</sub> concentration in seawater, operating in the range 0-2000 ppm and with a declared accuracy equal to  $\pm 0.5\%$  of the maximum range (i.e.  $\pm 10$  ppm). Before each use, the probe was checked at ENEA by measuring a mixture of known CO<sub>2</sub> concentration, fluxed in air at ambient pressure for about half an hour. The reported standard uncertainty for the check was calculated by combining the contributions of the declared accuracy term and the measured repeatability, respectively. As a result, the metrological check was found to be reasonably verified. The second step consisted of an *in situ* activity, the metrological comparison between the standard ENEA probe and another one, mounted on a trimaran (Maserati Multi70). The system was a “FerryBox” type designed for racing yachts, which acquired physico-chemical data such as *p*CO<sub>2</sub>, temperature and conductivity of the seawater sampled in real-time by a special duct positioned at a depth of 1 m along the route followed by the trimaran. The tested *p*CO<sub>2</sub> analyzer was a LI-COR model LI-850, having a declared accuracy of  $\pm 0.5$  ppm. The *p*CO<sub>2</sub> data were referenced with a standard CO<sub>2</sub> gas mixture (the system performed an automated *p*CO<sub>2</sub> calibration every 24 hours). The results obtained from this comparison were satisfactory and are reported in [16].

## V. CONCLUSIONS

The outcomes of the MINKE project in the field of *p*CO<sub>2</sub> measurement and assessment of the data quality highlighted several challenges that are still open and need to be further addressed in the near future. The cooperation and networking among oceanographic institutes and NMIs represents a fundamental aspect in this context.

The metrological coherence of the measurement models and the metrological traceability of the four EOVS involved in the carbonate system must be investigated and the corresponding uncertainties quantified. The possible routes of traceability of each variable should be investigated, performing respective experiments, and assessing their coherence. The two case studies presented are examples of application of the metrological principles to support the metrological traceability of measurements results obtained for the variable *p*CO<sub>2</sub> in the field.

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