

MARICAL field inter-calibration exercise

Report of the field inter-calibration on sampling and analytical procedures for Estonian, Finnish and Swedish institutes carrying out HELCOM monitoring

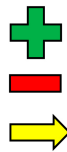


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Summary

Estonian, Finnish and Swedish institutes carrying out national HELCOM Baltic Sea monitoring programmes participated in a field inter-calibration for parameters describing eutrophication.



Positive results for nutrients – this is the main thing.

Not so much for chlorophyll a and dissolved oxygen.

Additional comparisons for silica, pH and CTD-hydrography.

It is suggested that the HELCOM parties could include this kind of field inter-calibrations, either between the neighbouring countries or as a basin-wide project, in their annual monitoring programmes.

Introduction

The EU aims at having the European seas in a good environmental condition or establishing them as such. In the process, all the HELCOM parties have committed to manage their sea areas according to the HELCOM Baltic Sea Action Plan (BSAP, HELCOM 2007) that is built on the implementation of the ecosystem-based approach. In this approach, national environmental monitoring programmes together deliver information that bridges up the pressures laid onto the environment with their impacts. This way, monitoring of the sea forms the foundation of marine management; we cannot plan effective programmes of measures without knowing the current state of the environment and where it is heading to.

Shared work

Monitoring responsibility in the Baltic Sea area is divided by the riparian countries according to their exclusive economic zones. The joint monitoring system meets, in principle, the requirement of the EU Marine Strategy Framework Directive (MSFD, EU 2008), that is a consistent coordinated approach for the Baltic Sea. The HELCOM monitoring and assessment strategy states that "The countries bordering a sub-basin should coordinate their monitoring cruises and make arrangements for e.g. taking turns in sampling certain areas or sharing responsibilities of monitoring of certain parameters with the idea that monitoring methods are harmonised...". This practically means that whenever the basin-wide monitoring is carried out by more than one HELCOM party, they should try to verify that the collected information is in line with each other. This, however, has not been done on a routine basis.

For chemical oceanography, all harmonizing work that has been done has centred in participating commercial proficiency tests without clear guidelines for when or how. These tests probe the analytical performance of an environmental laboratory, and by ignoring the quality of the field work, tell little about the HELCOM party's true monitoring performance.

Concentrating on routine work

Estonia, Finland and Sweden carried out a field inter-calibration targeting to clarify the quality of the monitoring data delivered by the Estonian, Finnish and Swedish institutes that are responsible for carrying out the national HELCOM Baltic Sea monitoring programmes. The target was the parameters that are employed in eutrophication assessments. The participants were instructed to carry out sampling at the monitoring station and consequent laboratory analyses as routinely as possible to yield insight into true monitoring performance.

The blueprint of this inter-calibration was created in the context of the Gulf of Finland Year 2014. Then, the field inter-calibration was carried out between Estonia, Finland and Russia. That project was the first of its kind and a success and provided the outline for this study.

The participating institutes

The participating institutes / contacting persons were:

- EST: Estonian Marine Institute (EMI), University of Tartu / Andres Jaanus
- EST: Marine Systems Institute (MSI), Tallinn University of Technology / Urmas Lips
- FIN: Finnish Environment Institute (SYKE) / Mika Raateoja
- FIN: Finnish Meteorological Institute (FMI) / Tero Purokoski
- SWE: Swedish Meteorological and Hydrological Institute (SMHI) / Johan Håkansson

In Finland, the monitoring responsibility is divided between FMI (marine physics) and SYKE (marine chemistry). Here, FMI was responsible for the CTD-data flow and SYKE for chemical analyses.

At all institutes, the analytical methods for the parameters under comparison were validated to the brackish water matrix. The testing environments of all the institutes were accredited by the national accreditation body in general, and except for hydrogen sulphide for MSI, the analytical methods were accredited specifically for the brackish water matrix as well.

Inter-calibration

The inter-calibration was conducted at the Estonian monitoring station F3 in the halfway between Helsinki and Tallinn in the Gulf of Finland, Baltic Sea (59°50.301'N, 24°50.298'E, nominal depth 80 meters) on 11 September 2019. FMI, SMHI and SYKE entered the station on board R/V "Aranda" while EMI and MSI did this onboard R/V "Salme".

The vessels stayed at the station approximately 100 meters apart, thus guaranteeing in its part that the results describe as much the same offshore water mass as possible. The significance of this source of error was assessed during the Gulf of Finland Year 2014 inter-calibration (Raateoja 2014). Then, it was concluded that this source of error was insignificant compared to the uncertainty stemming from the analytical determination.

The inter-calibration had the agreed timetable but high seas on that day forced the vessels not to follow it to the letter. Moreover, FMI and SMHI used separate CTD / Rosette systems on board R/V "Aranda". An inevitable time lag resulted as the FMI's rosette had to be changed to the SMHI's one. The water sampling was done within a 4-hours and the CTD casts within a 3-hours time window.

Scope of the study

This study was not a proficiency test by nature where the obtained results are compared against a specific nominal value (considered as "true value"). The nominal value is basically estimated as the average value of the result pool reported by the participants. Here, a pool of four individual results did not create a reliable statistical basis for applying any nominal value as a standpoint for comparison.

A consequence of the lack of "true value" is the descriptive characteristics of the study. As there was no nominal value there was no way to judge whether a participant performed better or worse than some other. We merely reported the discrepancies.

Another characteristic of the study stems from its real-life viewpoint. This report is not based on a strict scientific protocol, but rather, utilizes science in a degree that is needed to reach the primary target; to reveal discrepancies in the data that make a difference in the scale of marine management. In short, broad lines are preferred to small features.

This study reports the observed discrepancies but does not try to explain them by, e.g., methodological differences. To name a few, pH was measured either in *in situ* temperature or at 25° C and then corrected for *in situ* temperature, depending on a participant. Furthermore, the handling of the samples (particularly freezing) varied between the participants. Again, density was expressed as variants: Sigma-t, Sigma-theta or Sigma O. Addressing mathematical/technical/analytical reasons behind the differences in the monitoring performance requires a controlled target-oriented project with sound statistics based on numerous observations and is not within the scope of this study.

Water chemistry

The scope of the inter-calibration was the parameters that indicate eutrophication either directly or indirectly, forming the basis for the HELCOM's eutrophication assessment of the Baltic Sea. The parameters were: phosphate (PO_4), total phosphorus (TTP), ammonium (NH_3), nitrite (NO_2), the added nitrite and nitrate ($\text{NO}_2 + \text{NO}_3$), total nitrogen (TOTN), dissolved oxygen (O_2) and chlorophyll *a* (Chl *a*). These parameters provide the raw data for the calculation of six – out of a total of nine – HELCOM's core indicators for eutrophication at the time of writing this report [chlorophyll-*a*, dissolved inorganic nitrogen (DIN), dissolved inorganic phosphorus (DIP), total phosphorus (TP), total nitrogen (TN), oxygen debt]. Additionally, silicate, pH and hydrogen sulphide (H_2S ; a proxy for oxygen demand, "negative" oxygen) were compared. The sensitivity and precision of the methods as reported by the participants can be found in Table 1.

Surprisingly, the station F3 was oxic at the time of the visit, so H_2S could not be tested. Furthermore, NO_2 levels were near or below the limit of quantification, depending on the laboratory, and consequently its testing was unreasonable. The latter was not a problem as NO_2 is already part of $\text{NO}_2 + \text{NO}_3$.

Sampling followed the monitoring guidelines of the HELCOM coordinated monitoring program; 1, 5, 10, 15, 20, 30, 40, 50, 60, 70 and 79 meters. Due to harsh conditions (strong southerly wind up to 16 m/s and significant wave height of > 2 meters) the sampling on board R/V "Salme" had to be restricted to those depths where replicate samples were taken at. Therefore, EMI could not collect samples from 15, 30, 40 and 79 meters, and MSI from 1, 5, 15, 30, 40 and 79 meters.

The institutes differed in a way they handled the samples. MSI, SMHI and SYKE did not freeze samples except the Chl *a* sample after filtration. EMI froze the samples except the ones for most labile parameters (NH_3 , pH and O_2). The throughput times of the laboratories can be found in Table 2.

With the lack of any nominal value as a standpoint for comparison the best option to go forward was to put an individual result into perspective by using the reported measurement uncertainty (U) linked to the result. An average of the U values reported by the participants was used as a first-order metrics to identify possible questionable features in the dataset. The criterion was set as follows; if the deviation was smaller than the average U the result was satisfactory, otherwise the result was questionable. For comparisons, all results below the limit of quantification were excluded.

Table 1. Details for analytical performance, as reported by the participants

Note: U = expanded measurement uncertainty, LOQ = Limit of quantification, nr = not reported

Analytical parameter	Test parameter	EMI	MSI	SMHI		SYKE
PO ₄	U	24%	16%	12% if < 0.2 µmol/l	0.05 µmol/l if < 0.5 µmol/l	
				4% if > 0.2 µmol/l	10% if > 0.5 µmol/l	
	LOQ (µmol/l)	0.03	0.06	0.02	0.14	
TOTP	U	16%	11%	28%	0.07 µmol/l if < 1 µmol/l	
					15% if > 1 µmol/l	
		0.16	0.19	0.30	0.14	
NH ₃	U	25%	26%	7%	0.10 µmol/l if < 1 µmol/l	
					15% if > 1 µmol/l	
	LOQ (µmol/l)	0.14	0.14	0.20	0.10	
NO ₂	U	15%	nr	12% if < 0.2 µmol/l	0.04 µmol/l if < 0.4 µmol/l	
				4% if > 0.2 µmol/l	10% if > 0.4 µmol/l	
	LOQ (µmol/l)	0.02	nr	0.02	0.08	
NO ₂ + NO ₃	U	22%	14%	7% if < 1.5 µmol/l	0.15 µmol/l if < 1 µmol/l	
				4% if > 1.5 µmol/l	15% if > 1 µmol/l	
	LOQ (µmol/l)	0.07	0.07	0.10	0.30	
TOTN	U	18%	11%	14%	2 µmol/l if < 10 µmol/l	
					15% if > 10 µmol/l	
	LOQ (µmol/l)	0.36	2.4	5.0	3.2	
Silica	U	31%	7%	8% if < 5 µmol/l	10 %	
				3% if > 5 µmol/l		
	LOQ (µmol/l)	0.04	0.13	0.10	0.50	
H ₂ S	U	33%	nr	9%	30 %	
	LOQ (µmol/l)	0.50	0.50	4.0	1,1	
pH	U (at pH 7)	2%	2%	9%	1 %	
	LOQ (unitless)					
O ₂	U	14%	7%	6%	0.30 ml/l if < 4 ml/l	
					6% if > 4 ml/l	
	LOQ (ml/l)	0.00	0.07	0.10	0.36	
CHL α	U	15%	26%	20% if < 1 µg/l	39%	
				12% if > 1 µg/l		
	LOQ (µg/l)	0.10	0.50	0,20	0.90	

Replicates

Three replicates were collected in order to have an idea of the repeatability of the analytics. Replicate depths were as follows: 50 meters for nutrients; 5 meters for O₂; 20 meters for pH; and 10 meters for Chl *a*.

The repeatability was estimated with the coefficient of variation (%) that equals the standard deviation of the replicates divided by the average of the replicates. No specific threshold value for good performance was given, rather, the participants were compared to each other in relative terms.

Table 2. The time interval between sampling and analysis in this study

Analytical Parameter	EMI	MSI	SMHI	SYKE
PO ₄	27 days	20 hours	3 hours	1 hour
TOTP	28 days	20 hours	20 days	3 hours
NH ₃	2 days	20 hours	3 hours	2 hours
NO ₂ +NO ₃	27 days	20 hours	3 hours	1 hour
TOTN	28 days	20 hours	20 days	3 hours
Silica	26 days	20 hours	3 hours	1 hour
pH	< 1 hour	< 1 hour	1 hours	1 hour
O ₂	< 1 hour	< 1 hour	3 hours	2 hours
Chl <i>a</i>	15 days	7 days	15 days	6 days

CTD-data

CTD-data was not in the core of this project. Thus, there was no test parameter for the CTD-data and the comparison was descriptive. The testing approach was the average of the pressure interval db +/- 0.5 db (i.e., 1-meter averages) for water temperature, salinity, density, O₂, and a fluorescence-based proxy for algal biomass.

EMI, MSI and SMHI reported their CTD-based *in vivo* chlorophyll *a* fluorescence yields as actual Chl *a* concentration. As FMI / SYKE did not do this conversion, their *in vivo* chlorophyll *a* fluorescence yield was not directly comparable to the others.

Outcome

Environmental monitoring forms the basis for the management of the Baltic Sea by providing the starting point; it ensures that the criteria for a good ecological state of the Baltic Sea are based on facts. National programmes of measures taken place both in the sea and the catchment are employed to reach this good ecological state. Subsequent state assessments tell us how well programmes of measures have managed.

Environmental monitoring can thus be regarded as a fundamental tool for marine management. Its effectiveness is however dependent on the quality of the sampling / analytical work. A high-quality level of the environmental information is at least as important as having this information in the first place. An estimate of eutrophication parameter that is clearly erroneous may lead misinterpretations in the state assessments, and hence, have far worse consequences than has no estimate at all.

Quality control related work within environmental monitoring aims at providing a situation where far-reaching environmental decisions are based on reliable target setting and state assessments that in turn are based on a precise and reliable data flow. It is to note that within the northern Baltic Sea countries the level of quality assurance of the monitoring-related sampling/analytical protocol is generally regarded to be very good. Still, the countries' routine performance on a real-life condition is thus far poorly investigated.

Water samples

HELCOM's eutrophication assessment is based on the operational core indicators. The data flow for core indicators centres either entirely or for the main part, depending on an indicator, on the discrete water sample data collected at fixed monitoring stations. Hence, the water sample data forms the core of this comparison and is discussed in a context of each indicator for eutrophication.

The following aspects were ignored in the discussion: i) There seemed to be an issue in the sampling and/or handling of the MSI sample at 60 meters as evidenced by the nutrient data. ii) Variation at 20 meters as observed with many parameters was caused by local hydrography; chemical characteristics change rapidly in the vertical within the thermocline (20 meter sample located in the middle of it), and notable waving affected the actual sampling depth.

Chl *a*

HELCOM Chl *a* data originates from the upper 20 meters. Harsh conditions on the inter-calibration day forced the sampling on board R/V "Salme" to concentrate on the depths where replicates were taken at, that is, 10 meters.

For Chl *a*, U varied from 12 to 39%, depending on a participant and a concentration level. We used 25% as a guideline and found the overall variation to be questionable

(Annex 1).

SMHI and SYKE reported values that were generally further from each other than that. If all the 10 meters data are included in the comparison, SYKE seemed to report lower Chl *a* concentration than the others, although this conclusion has no basis more reliable than a single sample.

Dissolved inorganic nitrogen (DIN)

In the core indicator "dissolved inorganic nitrogen (DIN)", $DIN = NH_3 + NO_2 + NO_3$.

For NH_3 , U varied from 7 to 26%, depending on a participant and a concentration level. We used 15% as a guideline and found the overall variation to be satisfactory (Annex 1).

In the NH_3 level $> 2 \mu\text{mol/l}$ the differences were within limits. The difference grew larger in the NH_3 level $< 1 \mu\text{mol/l}$ but were still tolerable as the measurement uncertainty also grows at low concentrations.

For $NO_2 + NO_3$, U varied from 4 to 22%, depending on a participant and a concentration level. We used 15% as a guideline and found the overall variation to be satisfactory; the differences were generally within limits for $NO_2 + NO_3$.

Dissolved inorganic phosphorus (DIP)

For PO_4 , U varied from 4 to 24%, depending on a participant and a concentration level. We used 15% as a guideline and found the overall variation to be satisfactory; the differences were within limits for PO_4 (Annex 1).

Total nitrogen (TOTN)

For TOTN, U varied from 11 to 18%, depending on a participant and a concentration level. We used 15% as a guideline and found the overall variation to be satisfactory with an additional note (Annex 1).

The differences for TOTN were generally within limits. However, EMI reported values that were frequently lower than those of SMHI and SYKE.

Total phosphorus (TOTP)

For TOTP, U varied from 11 to 28%, depending on a participant and a concentration level. We used 20% as a guideline and found the overall variation to be satisfactory; the differences were within limits for TOTP (Annex 1).

Oxygen debt

For O_2 , U varied from 6 to 14%, depending on a participant and a concentration level. We used 10% as a guideline and found the overall variation to be questionable (Annex 1).

EMI and MSI provided higher estimates of O_2 than did SMHI and SYKE. Furthermore, SMHI and SYKE were in line with each other while EMI and MSI were not.

Silica

For silica, U varied from 3 to 31%, depending on a participant and a concentration level. We used 15% as a guideline and found the overall variation to be satisfactory with an additional note (Annex 1).

The differences for silica were generally within limits. EMI reported values that were somewhat lower than the others.

pH

For pH, U was reported to be 1, 1, 2 and 9% (calculated at pH 7), depending on a participant. Due to the logarithmic scale, the estimation of an average U is problematic. We used 3% as a guideline and found the overall variation to be satisfactory (Annex 1).

The results for pH were within generally limits. EMI reported somewhat lower values at 60 and 70 meters.

Replicates

Expectedly, the parameters, the analyses of which include a significant share of manual work (Chl *a*, O₂, pH) presented the highest variation within the replicates (Figure 1).

With respect to nutrients, the variation within the replicates was higher for EMI than for the others.

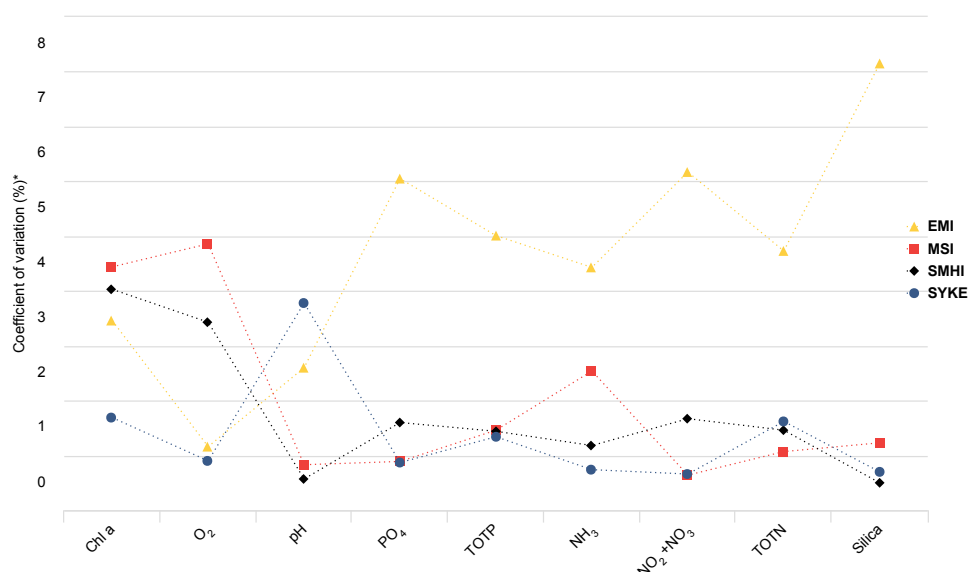


Figure 1. Variation (CV, %) in the replicates between the participants

*(100 x standard deviation of replicates / average of replicates)

CTD-data

FMI differed from the others in the commencement of the main density gradient co-occurring with the thermocline; temperature started to decrease 3 meters shallower, salinity started to increase 2–3 meters shallower and, as a result, density started to increase 3 meters shallower than for the others. Moreover, O₂ within the density gradient started to decrease 3–5 meters shallower than for the others (Annex 2). The shallower thermocline, halocline and oxycline were probably caused by temporal discrepancy in the CTD casts and a technical aspect. CTD-profiling and accompanying sampling for FMI / SYKE occurred earlier than those for SMHI, EMI and MSI. A strong wind and subsequent mixing on that day can explain a part of the difference; the results from the sampled parameters give back-up for this conclusion. Also, the pressure sensor of the FMI's CTD probably showed too low values and thus averaging created depth bins which were 1 dbar shallower when compared to other CTDs (T. Purokoski, personal communication).

SMHI reported somewhat higher temperature and salinity in the surface mixed layer while density reported by EMI increased relatively more with depth than it did with the others (Annex 2). The largest deviations were observed with the CTD-parameters that are dependent on the secondary calibration against water sample data. Quantitatively, the institutes reported O₂ gradients of a similar shape but at notably different level; EMI had the highest level, then MSI, SMHI and FMI. The same applied to the *in vivo* Chl *a* fluorescence yield converted to Chl *a* concentration. The general level in the surface mixed layer varied markedly. SMHI reported lower concentrations of Chl *a* than EMI and MSI. EMI did the same as compared to MSI although the difference was smaller. The baseline fluorescence yield below the thermocline that has little biogenic origin, varied relative to the levels observed in the surface mixed layer.

Synthesis

Estonian, Finnish and Swedish institutes that are responsible for national HELCOM Baltic Sea monitoring programmes carried out a field inter-calibration targeting to clarify the coherence of the eutrophication related monitoring data. The project was carried out in the field condition in order to include all the sources of error from sampling, sample handling and analytics. This way the true monitoring performance was revealed.

A project of this kind has basically two potential outcomes. The participants may perform satisfactorily in the tests which is of course the preferred outcome. However, this result leads to the business-as-usual continuation of the monitoring protocol, and thus misses this one-time chance to refine the sampling / analytical protocol. So, another option that seems to apply here – some room for development was recognized – is a valuable result too.

Water sample data

The level of coherence amongst the participants for water sample data:

- Chl *a*: questionable (SMHI and SYKE differed)
- DIN: satisfactory
- DIP: satisfactory
- TOTN: satisfactory with a note (Somewhat lower values for EMI)
- TOTP: satisfactory
- O₂: questionable (SMHI and SYKE differed from EMI and MSI)
- Silica: satisfactory with a note (Somewhat lower values for EMI)
- pH: satisfactory

The good news is that the causative feature in eutrophication, the resident nutrient quotas, was estimated coherently. This result ensures in its part a reliable data flow for marine management to mitigate eutrophication.

Algal biomass approximated by Chl *a*, being a direct consequence of eutrophication, seems to need further quality-related work. To get a more profound picture of Chl *a* a new comparison would actually be needed due to the lack of data in this round. The lack of data was due to adverse weather. A regular proficiency test could be the easiest option here. Still, some steps forward can be taken already based on these results.

Notable deviations were observed with dissolved O₂, too, that is an indirect

consequence of eutrophication.

CTD-data

The most striking feature in the CTD-data related to eutrophication was the tendency of the participants to report O₂ gradients of a similar shape but quantitatively at notably different level. Similarly, the conversion of *in vivo* chlorophyll *a* fluorescence yield to actual Chl *a* concentration resulted in notably different outcome amongst the participants. These indicate issues in the secondary calibration of data.

Outlook

As monitoring responsibility in the Baltic Sea area is divided by the riparian countries, the harmonisation of the employed sampling and analytical methods becomes crucial, influencing all subsequent steps in the marine data flow and data utilisation. This study provided the participants with a chance to take a critical look at their monitoring process keeping better data cohesion between the countries in mind, hopefully leading to an enhanced dialog.

A continuation of field projects of this kind, funded by the HELCOM parties, is suggested in order to secure the future coherence of the HELCOM monitoring data flow. Although this project concerned the northern Baltic Sea and its riparian countries this blueprint can be easily copied to other parts of the Baltic Sea; it could be arranged between the neighbouring countries or as a basin-wide project. The ideal situation would be if the parties could agree on and subsequently include this kind of field comparisons in their annual monitoring programmes. Routine work should be made better on a routine basis.

References

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Annex 1: Water sample figures

Figure A1.1. Chl *a* concentration. Arrow shows the average measurement uncertainty.

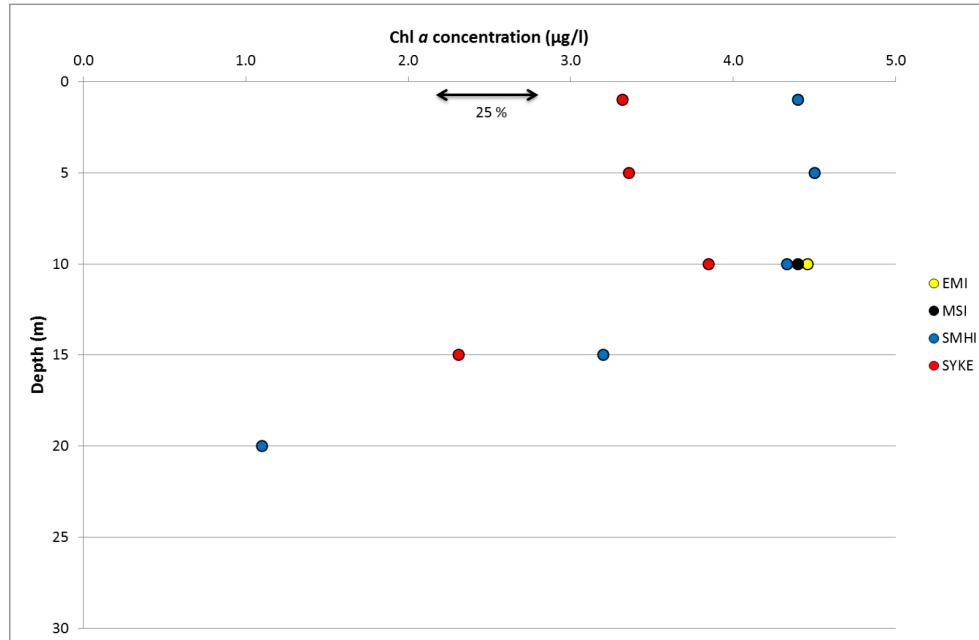


Figure A1.2. O₂ concentration. Arrow shows the average measurement uncertainty

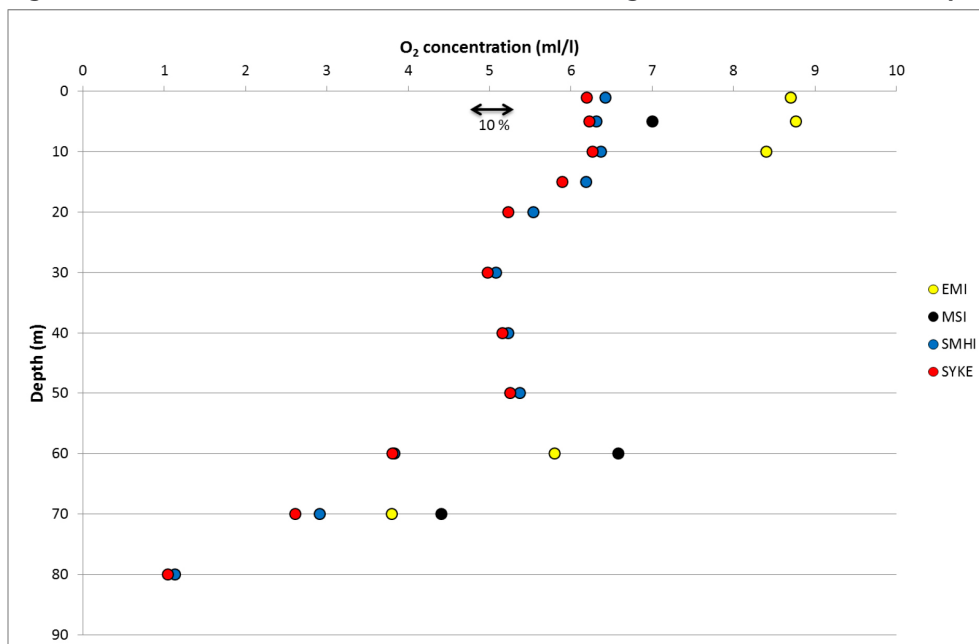


Figure A1.3. pH. Arrow shows the average measurement uncertainty

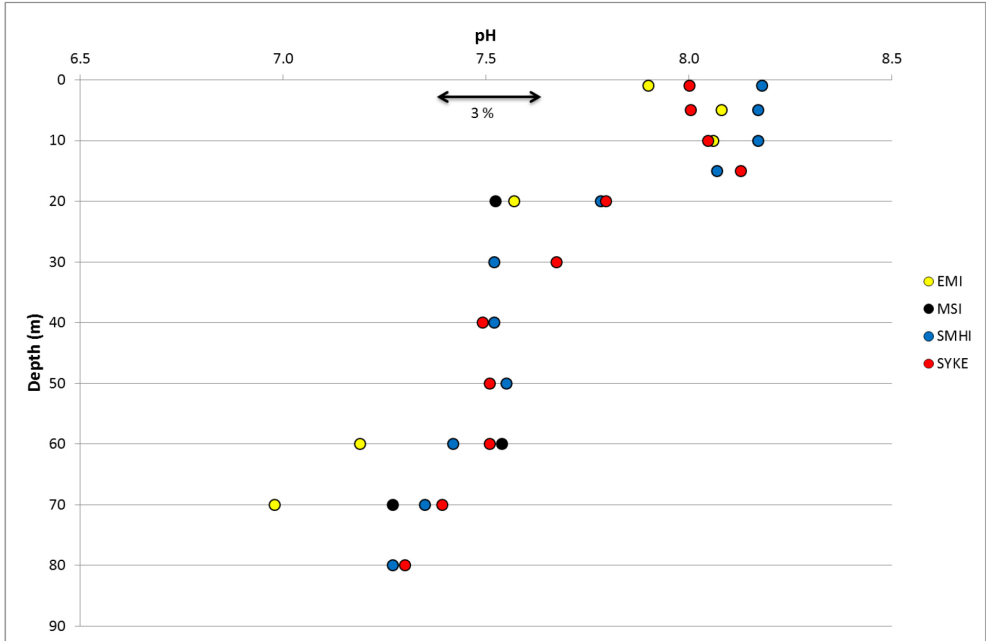


Figure A1.4. PO₄ concentration. Arrow shows the average measurement uncertainty

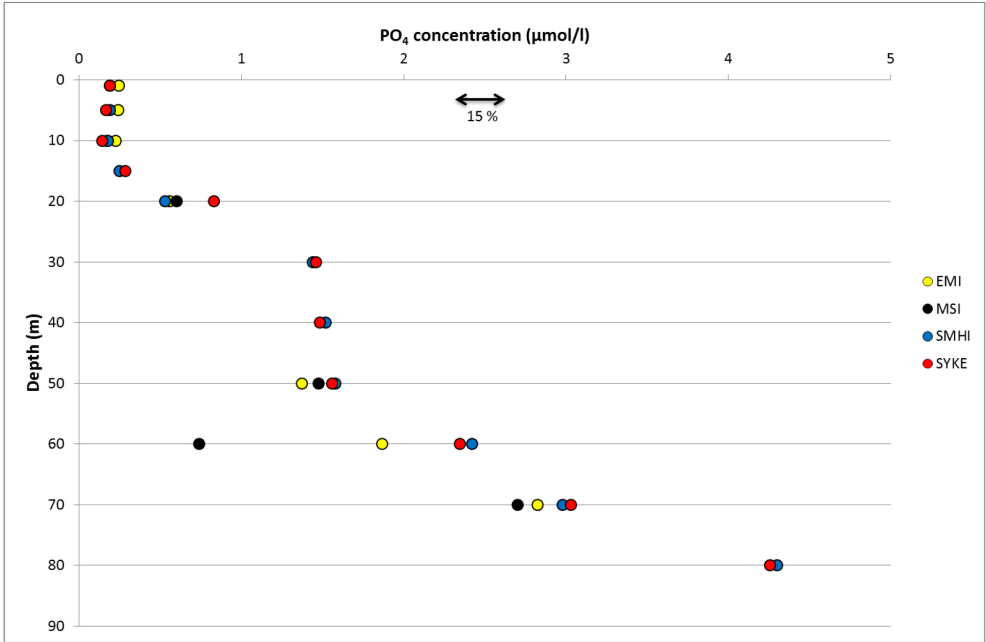


Figure A1.5. TOTP concentration. Arrow shows the average measurement uncertainty

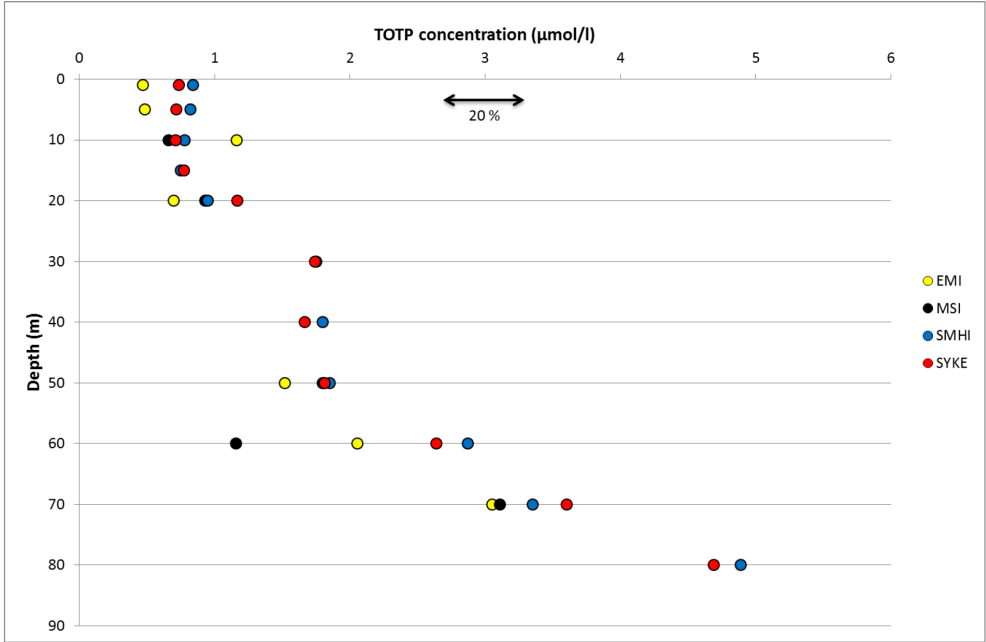


Figure A1.6. NH_3 concentration. Arrow shows the average measurement uncertainty

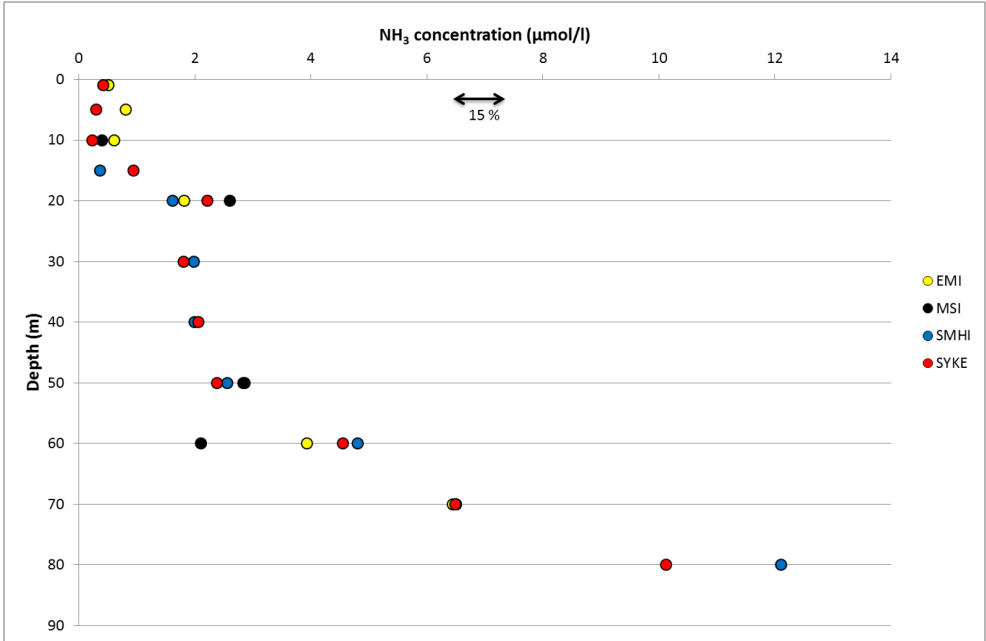


Figure A1.7. NO₂ + NO₃ concentration. Arrow shows the average measurement uncertainty

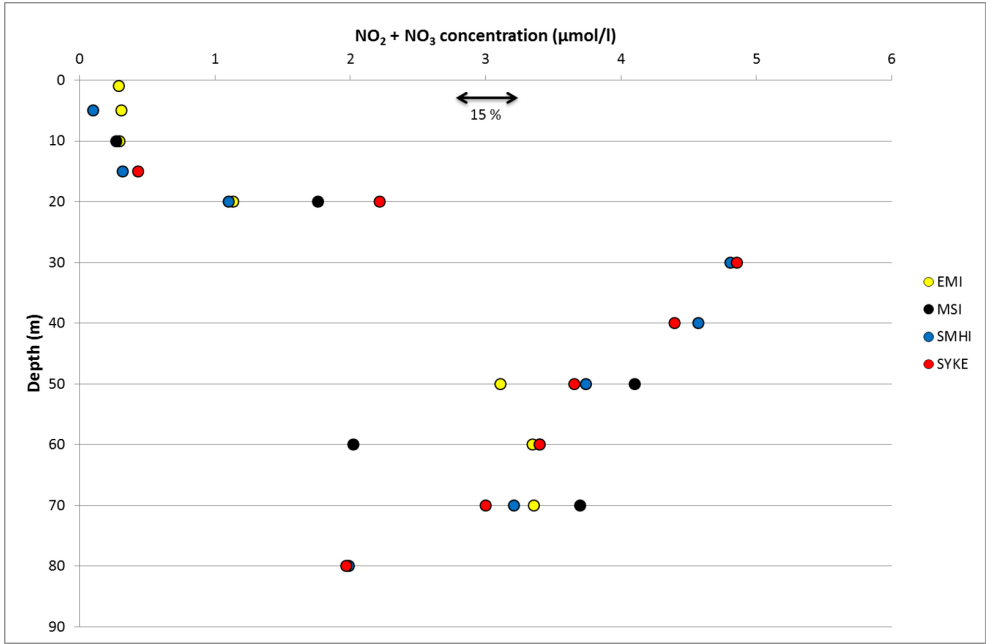


Figure A1.8. TON concentration. Arrow shows the average measurement uncertainty

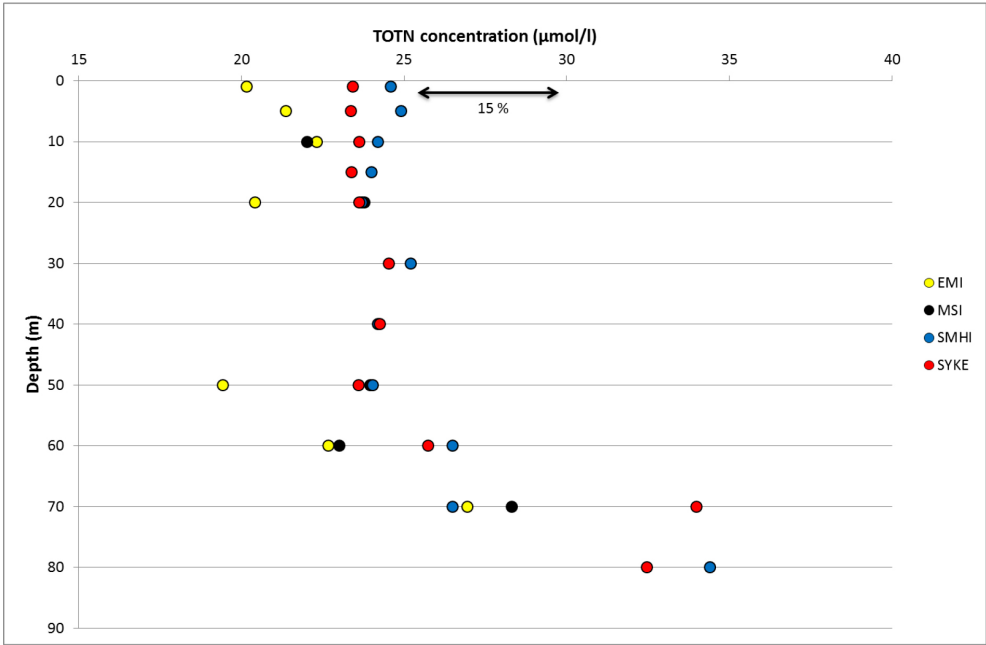
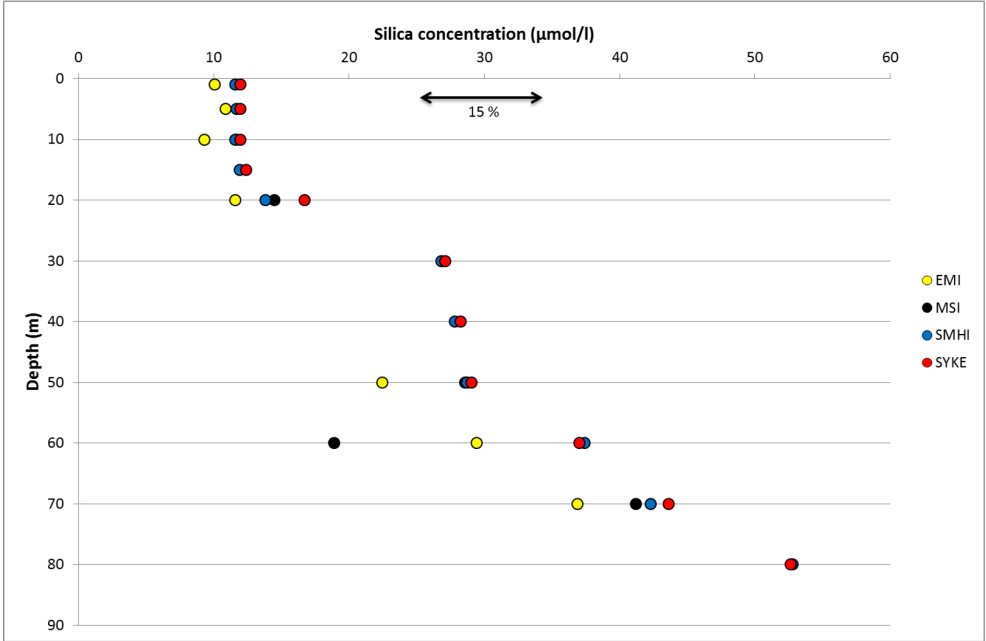


Figure A1.9. Silica concentration. Arrow shows the average measurement uncertainty



Annex 2: CTD-data figures

Figure A2.1. CTD-temperature

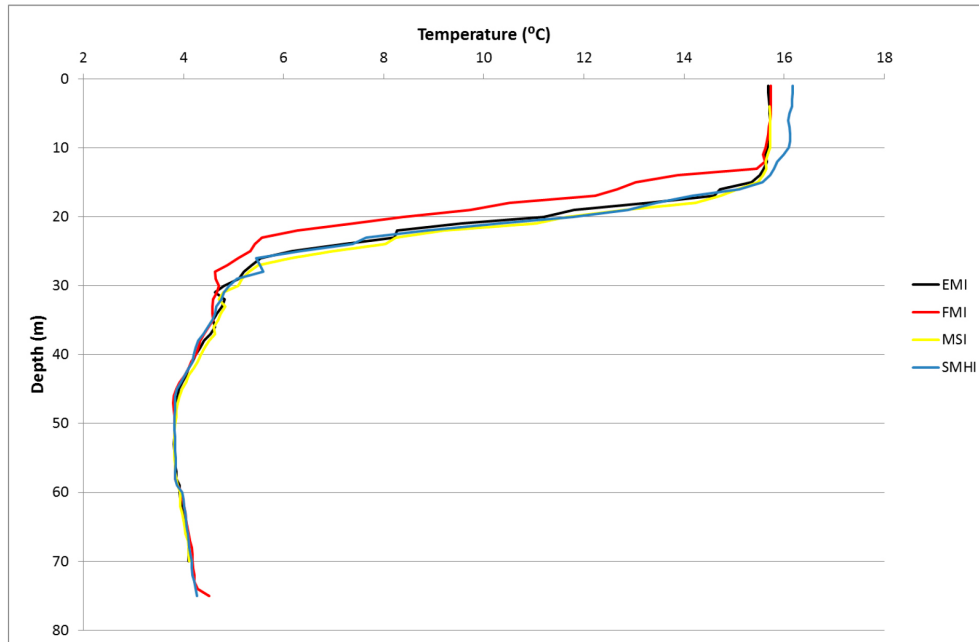


Figure A2.2. CTD-salinity

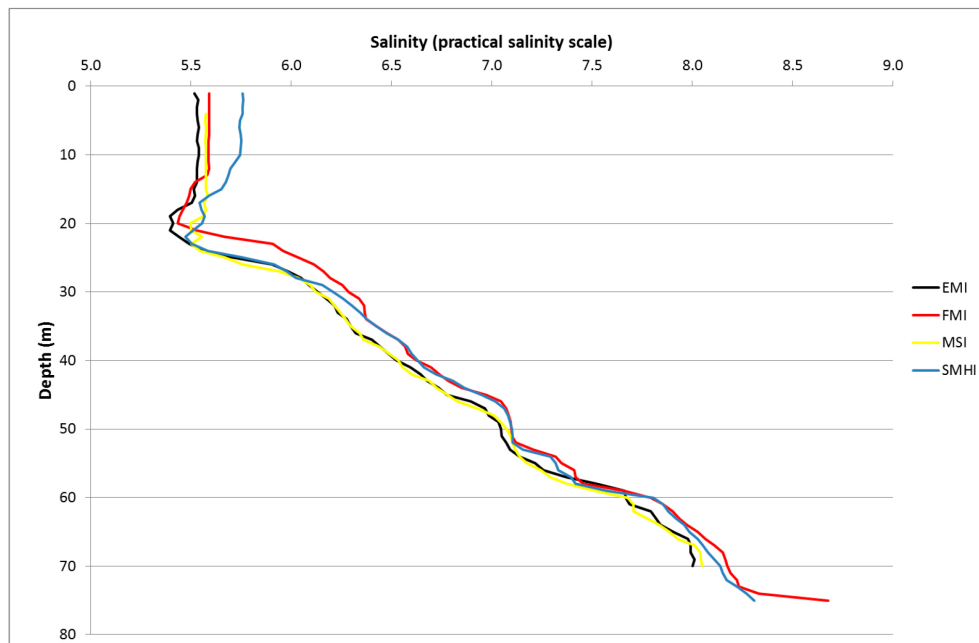


Figure A2.3. CTD-density

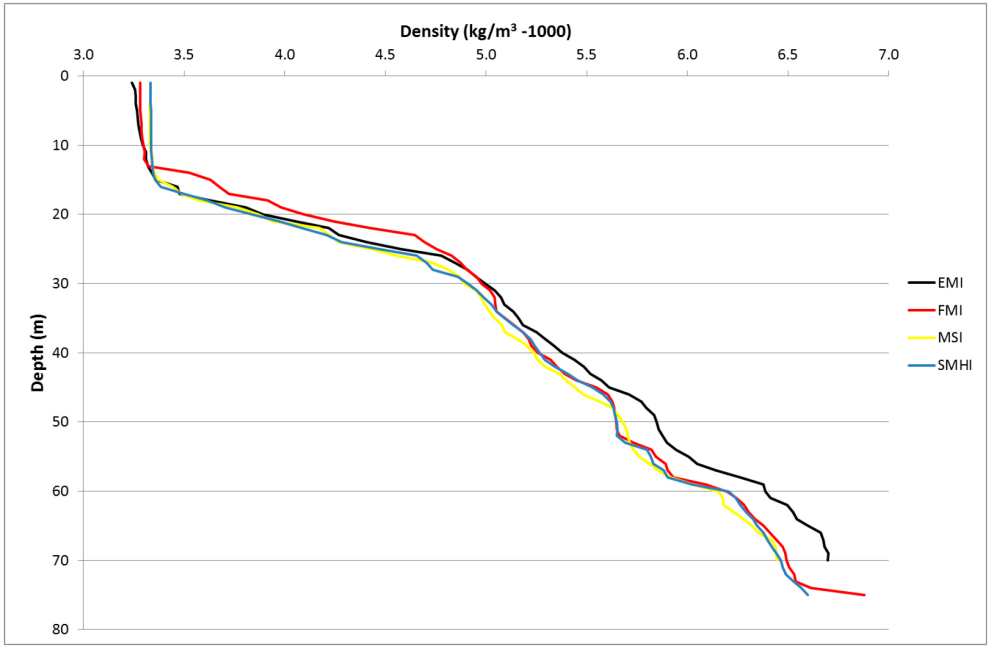


Figure A2.4. CTD-O₂ concentration

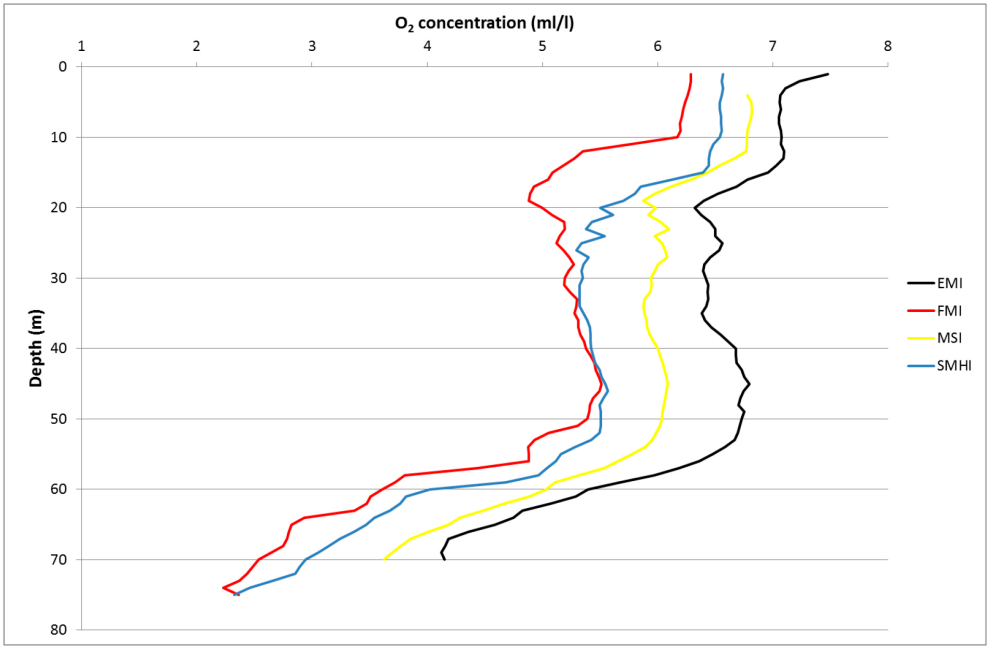
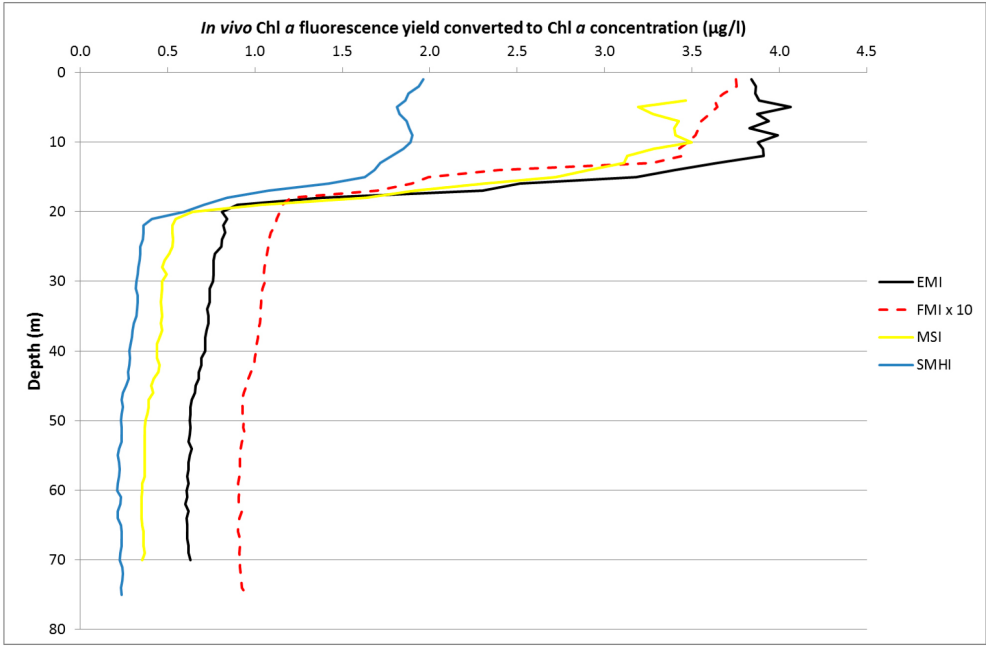


Figure A2.5. CTD Chl *a* concentration. FMI / SYKE presents *in vivo* fluorescence data



About this publication

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