RV *INVESTIGATOR*

HYDROCHEMISTRY DATA PROCESS REPORT

Voyage:	IN2018_v02
Chief Scientist	Tom Trull
Principal Investigator	Philip Boyd
Voyage title:	SOTS: Southern Ocean Time Series automated moorings for climate and carbon cycle studies southwest of Tasmania; Subantarctic Biogeochemistry of Carbon and Iron, Southern Ocean Time Series site
Report compiled by:	Peter Hughes & Julie Janssens

Contents

1 Executive Summary

The quality of the hydrology data is good.

Water samples collected during the voyage were assayed in the ship's hydrochemistry laboratory for nutrients, dissolved oxygen, and their salinity measured. The samples came from deployments of the CTD rosette and trace metal rosette as well as from experiments run by the science party.

Results for nutrient samples from experiments issued to the science parties during the voyage.

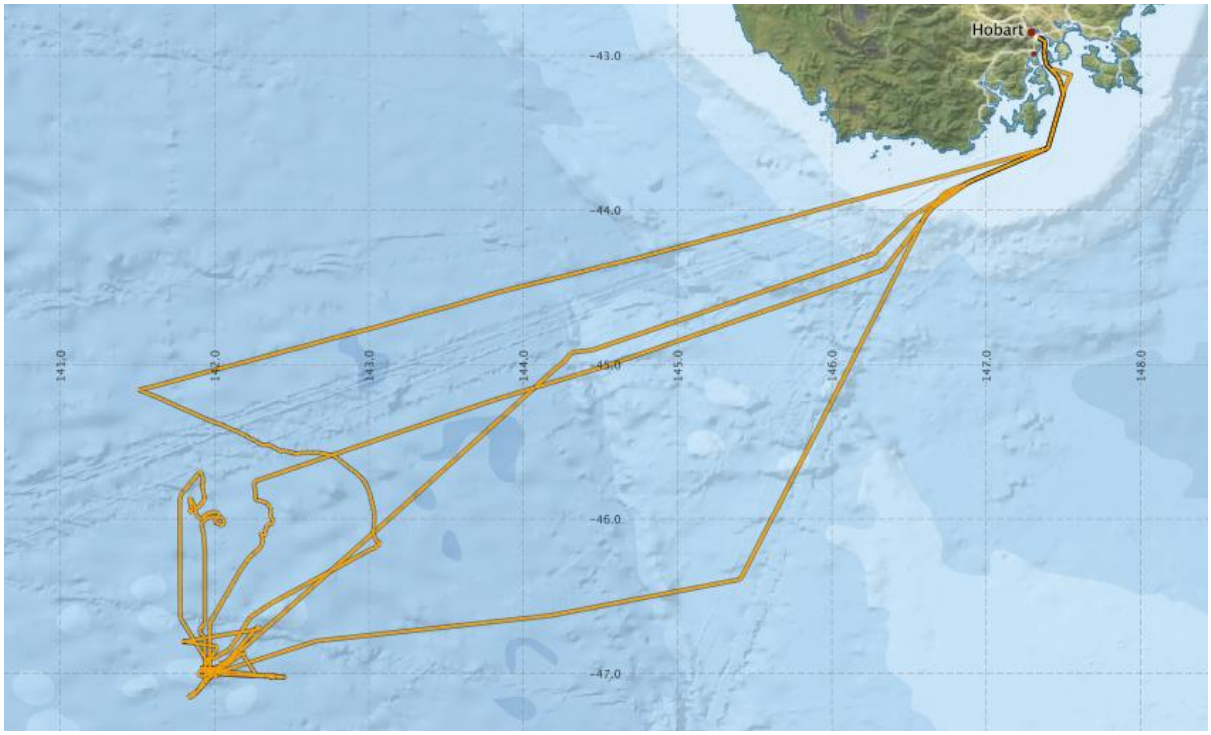
Final hydrology data, analytical methods, and related log sheets and processing notes can be obtained from the CSIRO data centre.

Contact: DataLibrariansOAMNF@csiro.au

2 Itinerary

Hobart to Hobart, March 3rd - 21st, 2018.

Voyage Track:



3 Key personnel list

Name	Role	Organisation
Tom Trull	Chief Scientist	CSIRO & ACE CRC
Lisa Woodward	Voyage Manager	CSIRO
Peter Hughes	Hydrochemist	CSIRO
Julie Janssens	Hydrochemist	CSIRO

4 Summary

4.1 Sample Type and Number Assayed

Analysis (instrument)	Number of Samples
Salinity (Guildline Salinometer)	85 CTD 48 TMR
Dissolved Oxygen (automated titration)	85 CTD
Nutrients (Seal AA3HR)	94 CTD 98 TMR 244 EXP

4.1.1 CTD

- Sampling point, 36 bottle rosette with 12L Ocean Test Equipment bottles deployed at depth for water collection.
- 7 CTD deployments in total. Deployments 2 thru 6 sampled and assayed by hydrochemistry.

4.1.2 TMR

- Sampling point, 12 bottle trace metals rosette.
- 5 deployments in total. Sampled by the trace metals team, assayed by hydrochemistry.

4.1.3 EXP

- Prepared and sampled by the science groups conducting the experiments, assayed by hydrochemistry.

4.2 Data Processing Overview

The sample meta-data, measured bottle salinity results, dissolved oxygen assay results and the nutrient assay raw data are processed by the CSIRO program HyPro. The final output is the hydrology data set. An overview of this process is illustrated in figure 1.

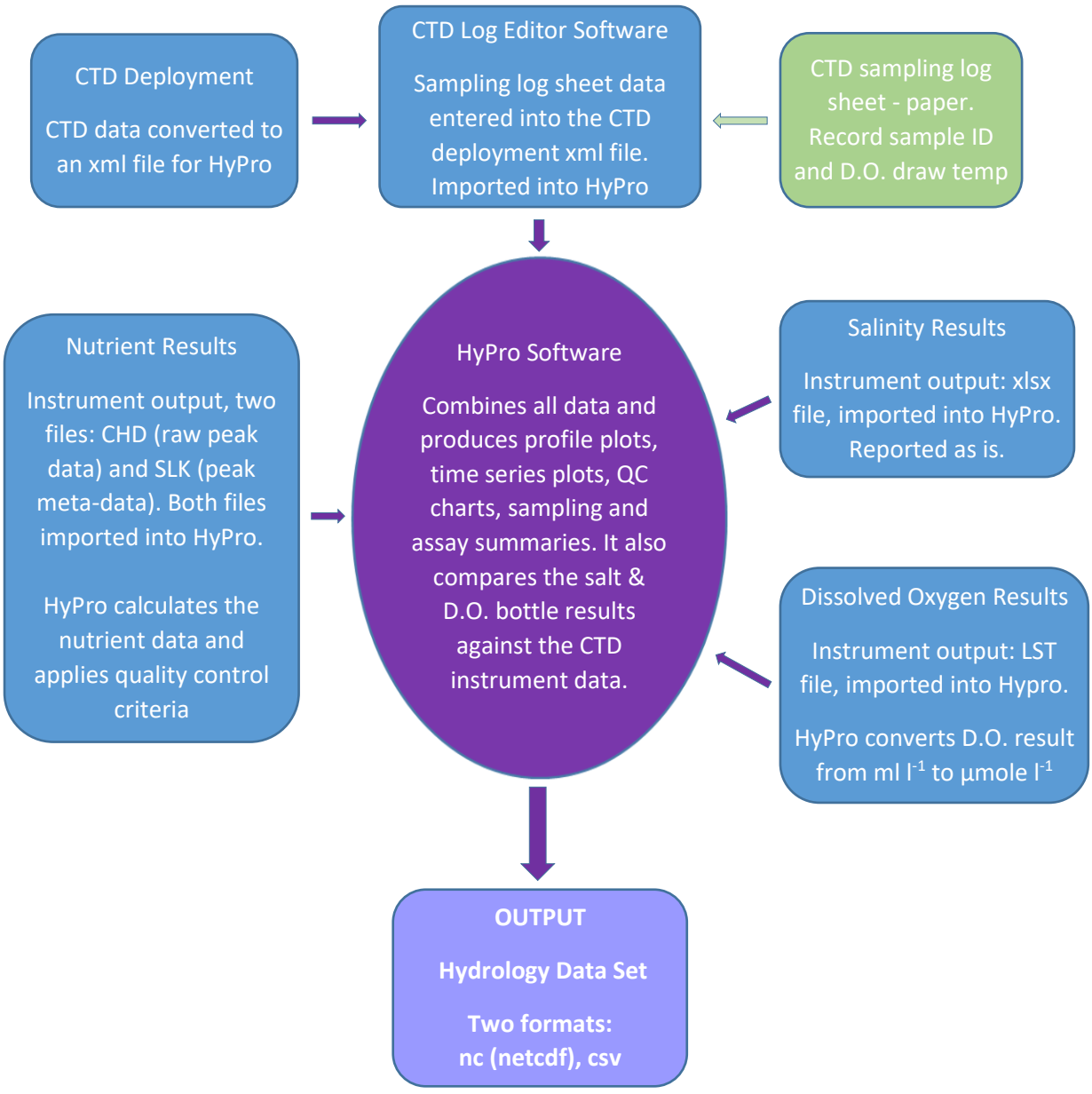


Figure 1: Hydrology Data Processing Flow Diagram.

5 Salinity Data Processing

5.1 Salinity Parameter Summary

Details	
CSIRO HyPro version	5.4
Instrument	Guildline Autosal Laboratory Salinometer 8400(B) – SN 72151
Software	OSIL Data Logger ver 1.2
CSIRO Hydrochem Method.	Sampling: WI_Sal_002 Measurement: SOP006
Accuracy	± 0.001 practical salinity units
Analists	Peter Hughes, Julie Janssens
Lab Temperature (±0.5°C)	21 -23°C during analysis.
Bath Temperature	24.01°C
Reference Material	Osil IAPSO - Batch P161, use by 03/05/2020, K_{15} = 0.99987
Sampling Container type	200 ml volume OSIL bottles made of type II glass (clear) with disposable plastic insert and plastic screw cap.
Sample Storage	Samples stored in the Salt lab for a minimum of 8hrs before measurement.
Comments	None.

5.2 Salinity Method

Salinity samples are measured on a high precision laboratory salinometer (Guildline Autosal 8400B) which is operated in accordance with its technical manual.

Practical salinity (S), is defined in terms of the ratio (K_{15}) of the electrical conductivity measured at 15°C 1atm of seawater to that of a potassium chloride (KCl) solution of mass fraction 32.4356 x 10^{-3}.

Before each batch of sample measurements, the Autosal is calibrated with standard seawater (OSIL, IAPSO) of known K_{15} ratio. A new bottle of OSIL solution is used for each calibration.

Method synopsis: Salinity samples are collected into 200ml OSIL bottles, filled from the bottom, via a PTFE straw, till overflowing. The bottle is removed from the straw and the sample is decanted to allow a headspace of approximately 25cm^3. A plastic insert is fitted, the bottle inverted and rinsed with water then capped and stored cap-down until measured. To measure, the Autosal cell is flushed three times with the sample and then measured after the fourth and fifth flush. Further flush-measurement cycles are done where the initial values are more than 3 digits different. The Osil Data logger software captures the conductivity ratio and calculates the practical salinity.

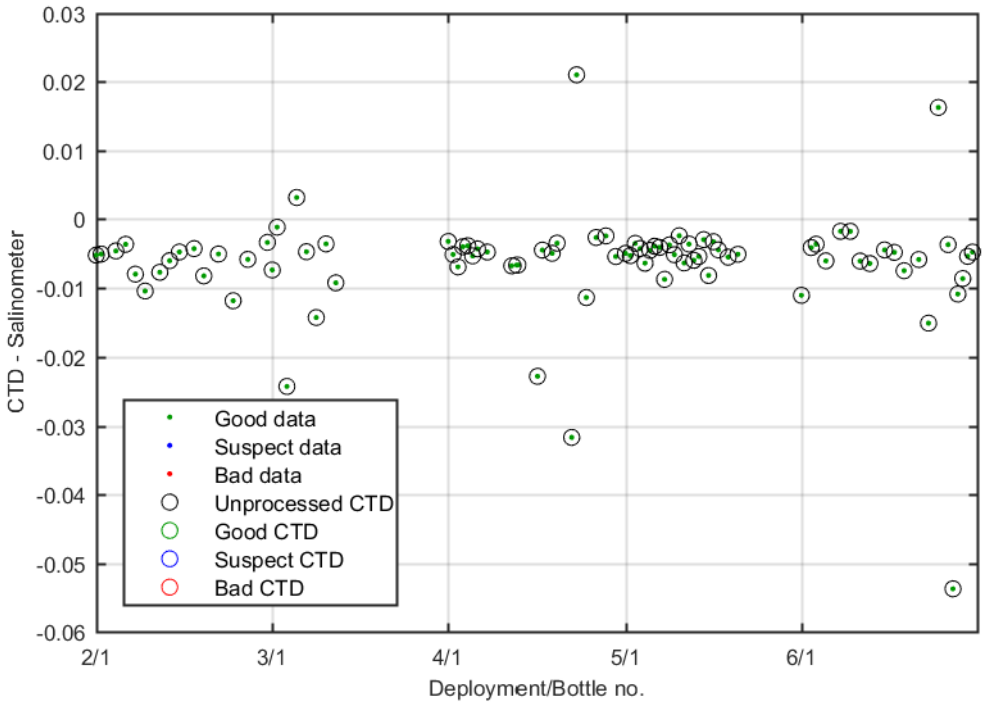
The output from the data logger software is imported into HyPro and collated with the CTD deployment meta-data.

5.3 CTD Salinity vs Bottle Salinity Plot

The difference between the unprocessed (uncorrected) CTD values and the measured bottle salinities is generally less than 0.01 PSU.

The unprocessed CTD values are adjusted (corrected) by DAP using the bottle results. The corrected values are not reported in the hydrology set. Please contact the DataLibrarians@csiro.au for corrected CTD data.

Note: dots = bottle samples, circles = CTD instrument (unprocessed)



5.4 Missing or Suspect Salinity Data

None.

6 Dissolved Oxygen Data Processing

6.1 Dissolved Oxygen Parameter Summary

Details	
CSIRO HyPro Version	5.4
Instrument	Automated Photometric Oxygen system (SIO)
Software	SCRIPPS
CSIRO Hydrochem. Method	Sampling: WI_DO_001 Assay: SOP005
Accuracy	± 0.5 µM
Analyst(s)	Peter Hughes & Julie Janssens
Lab Temperature (±1°C)	Variable, 20.0 - 23.0°C
Sample Container type	Pre-numbered 140 mL glass iodine determination flasks with glass stopper. 18 flasks per light-proof container.
Sample Storage	Samples stored in the hydrochemistry lab until analysis. All samples were analysed within ~48 hrs
Comments	None.

6.2 Dissolved Oxygen Method

SCRIPPS method used. The method is based on the whole-bottle modified Winkler titration of Carpenter (1965) plus modifications by Culberson *et al* (1991).

Method synopsis: The sample is collected in an iodine determination flask of known volume. 1mL of manganese (II) chloride solution followed by 1mL of alkaline iodide solution is added to the sample, the flask stoppered and inverted a minimum ten times. The dissolved oxygen oxidizes an equivalent amount of Mn (II) to Mn (IV) which precipitates. Just before titration, the sample is acidified, oxidizing the Mn (IV) back to the divalent state liberating iodine twice the original dissolved oxygen content of the sample. The tri-iodine is auto-titrated with a standardised thiosulphate solution using a Metrohm 665 Dosimat fitted with a 1ml burette. The endpoint is determined by measuring changes in the UV absorption of the tri-iodide ion at 365 nm. The point at which there is no change in absorbance is the endpoint.

Before each batch of sample assays, the thiosulphate solution is standardised by using it to titrate a 10ml aliquot of potassium iodate primary standard. A blank correction is also determined from the difference between two consecutive titres for 1ml aliquots of the same potassium iodate solution.

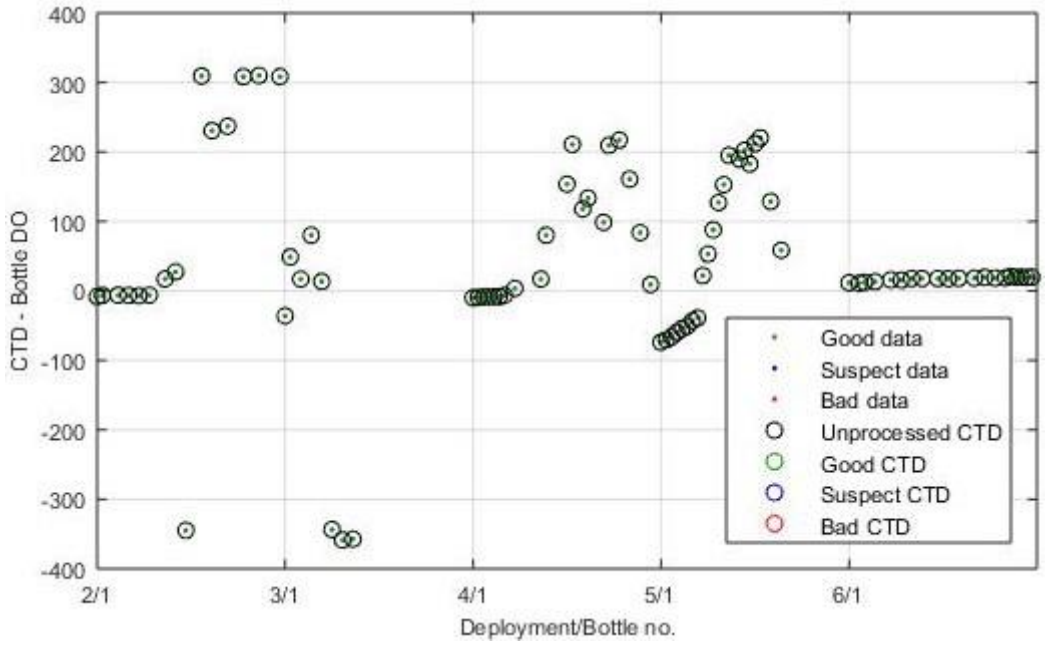
The output from the SIO instrument software is imported into HyPro and collated with the CTD deployment meta-data.

6.3 CTD Dissolved Oxygen vs Bottle Dissolved Oxygen Plot

The large discrepancy between the bottle values and the CTD instrument values is due to CTD processing issues during the voyage. The CTD values in this plot are unprocessed raw data.

The unprocessed CTD values are adjusted (corrected) by DAP using the bottle results. The corrected values are not reported in the hydrology set. Please contact the DataLibrarians@csiro.au for corrected CTD data.

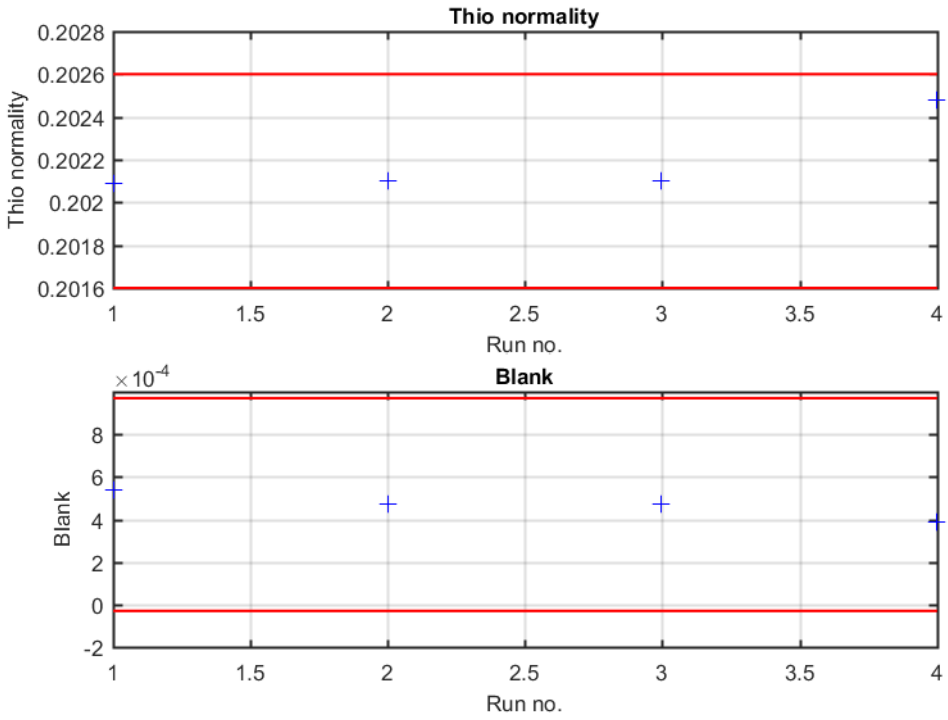
Note: dots = bottle samples, circles = CTD instrument (unprocessed)



6.4 Dissolved Oxygen Instrument titrant: thiosulphate normality and blank correction.

The normality of the thiosulphate titrant (0.2N) varied less than 0.0004 for all dissolved oxygen sample titrations. The blank correction is less than 0.0006mL.

For reference, titre volumes for dissolved oxygen bottle samples lay in the range 0.46 to 0.80 ml.



6.5 Missing or Suspect Dissolved Oxygen Data.

CTD	RP	Run	Flag	Reason for Flag or Action
5	15	2	141 (nc file)	No result. Operator error. Sample collected and assayed. Not stirred during titration thus endpoint absent.

7 Nutrient Data Processing

7.1 Nutrient Assay Parameter Summary

Details					
CSIRO HyPro version	5.4				
Instrument	Seal AA3HR				
Instrument Software	Seal AACE 6.10				
CSIRO Hydrochem. Method, sampling	WI_Nut_001				
CSIRO Hydrochem. Method, nutrient	SOP001	SOP002	SOP003	SOP003	SOP004
Nutrient	Silicate	Phosphate	Nitrate + Nitrite	Nitrite	Ammonium
Concentration range	112 µM	3.0 µM	42 µM	1.4 µM	2.0 µM
Method Detection Limit (MDL)	0.2 µM[1]	0.02 µM	0.02 µM	0.02 µM	0.02 µM
Matrix Corrections	none	none	none	none	none
Analysts	Julie Janssens, Peter Hughes				
Lab Temperature (±1°C)	Variable, 21 – 23°C				
Reference Material	KANSO, RMNS lot BW				
Sampling Container type	CTD: 50ml HDPE with screw cap lids. MTR and EXP: 12ml PP tubes with screw cap lids.				
Sample Storage	< 2 hrs at room temperature or ≤ 12 hrs @ 4°C				
Pre-processing of Samples	CTD and TMR: None. EXP: as prepared by the science parties.				
Comments	TMR and EXP samples collected by the science teams.				

7.2 Nutrient Methods

Nutrient samples are assayed on a Seal AA3HR segmented flow auto-analyser fitted with 1cm flow-cells for colorimetric measurements and a JASCO FP2020 fluorescence instrument as the ammonium detector.

Silicate (SOP001): colourimetric, molybdenum blue method. Based on Armstrong et al. (1967). Silicate in seawater is reacted with acidified ammonium molybdate to produce silicomolybdic acid. Tartaric acid is added to remove the phosphate molybdic acid interference. Tin (II) chloride is then added to reduce the silicomolybdic acid to silicomolybdous acid and its absorbance is measured at 660nm.

Phosphate (SOP002): colourimetric, molybdenum blue method. Based on Murphy and Riley (1962) with modifications from the NIOZ-SGNOS Practical Workshop 2012 optimizing the antimony catalyst/phosphate ratio and the reduction of silicate interferences by pH. Phosphate in seawater

forms a phosphomolybdenum complex with acidified ammonium molybdate. It is then reduced by ascorbic acid and its absorbance is measured at 880nm.

Nitrate (SOP003): colourimetric, Cu-Cd reduction – naphthylenediamine method. Based on Wood et.al (1967). Nitrate is reduced to nitrite by first adding an ammonium chloride buffer then sending it through a copper - cadmium column. Sulphanilamide is added under acidic conditions to form a diazo compound. This compound is coupled with 1-N-naphthly-ethylenediamine di-hydrochloride to produce a reddish purple azo complex and its absorbance is measured at 520 nm.

Nitrite (SOP003): colourimetric, naphthylenediamine method. As per nitrate method without the copper cadmium reduction column and buffer.

Ammonium (SOP004): fluorescence, ortho-phtaldiadehyde method. Based on Roger Kérouel and Alain Aminot, IFREMER (1997 Mar.Chem.57). Ammonium reacted with ortho-phtaldialdehyde and sulphite at a pH of 9.0-9.5 to produce an intensely fluorescent product. Its emission is measured at 460nm after excitation at 370nm.

SOP methods can be obtained from the CSIRO Oceans and Atmosphere Hydrochemistry Group.

7.3 HyPro Processing Parameters

All instrument parameters and reagent batches and operation events are logged for each analysis run. This information is available on request.

Result Details	Silicate	Phosphate	Nitrate + Nitrite	Nitrite	Ammonia
Data Reported as	µmol l^{-1}	µmol l^{-1}	µmol l^{-1}	µmol l^{-1}	µmol l^{-1}
Calibration Curve degree	Linear	Linear	Quadratic	Quadratic	Quadratic
# of points in Calibration	6	6	6	6	6
Forced through zero?	N	N	N	N	N
Matrix correction	N	N	N	N	N
Blank correction	N	N	N	N	N
Peak window defined by	HyPro	HyPro	HyPro	HyPro	HyPro
Carryover correction (HyPro)	Y	Y	Y	Y	Y
Baseline drift correction (HyPro)	Y	Y	Y	Y	Y
Sensitivity drift correction (HyPro)	Y	Y	Y	Y	Y
Data Adj for RMNS variance.	N	N	N	N	N
Medium of Baseline	18.2 Ω water. Dispensed from Milli Q				
Proportion of samples in duplicate.	<10%. CTD: Niskin fired at the greatest depth sampled in duplicate. Single samples collected for remaining depths.				
Comments	The reported data is not corrected to the RMNS. Per deployment RMNS data tabulated in appendix 8.2.				

7.4 HyPro Data Processing Summary

After a run, the raw absorbance/ fluorescence data is exported from the instrument and processed by HyPro. For each analyte, HyPro re-creates the peak traces, defines the region on the peak's plateau (peak window) used to determine the peak heights, constructs the calibration curve, applies corrections for carry-over, baseline and sensitive drifts then, derives the nutrient concentrations for each sample. The corrections are quantified using dedicated solutions included in every run.

HyPro uses criteria to identify suspect calibration points, noisy peaks, method detection limits that are above the nominal limit and, duplicate sample results that do not match.

With suspect calibration points, their contribution to the curve is given less weighting dependent on their distance from the final curve. The cut-off limits for good calibration data are:

- ±0.5% of the concentration of the top standard for silicate and nitrate+nitrite (as per WOCE).
- Within 0.02uM for phosphate, nitrite and ammonium.

HyPro classifies the quality of data as good, suspect or bad and flags accordingly. The flags are in the final hydrology data set. Flag key in Appendix 8.5.

7.5 Accuracy - Reference Material for Nutrient in Seawater (RMNS)

Japanese KANSO certified RMNS lot BW was assayed in triplicate in each run to monitor accuracy. The certified values are in table 1.

For in2018_v02, the majority of RMNS results are within 1% of their certified mean and within 0.02µM for nitrite. Phosphate is the exception with values within +2%. Plots of RMNS values for all runs are below.

The assayed RMNS values per CTD and TMR deployments are listed in appendix 8.2

The GO-SHIP criteria (Hyde *et al*., 2010), appendix 8.6, specifies using 1-3 % of full scale (depending on the nutrient) as acceptable limits of accuracy.

Table 1: RMNS lot BW concentrations with expanded uncertainty (µmol l^{-1}) at 21°C

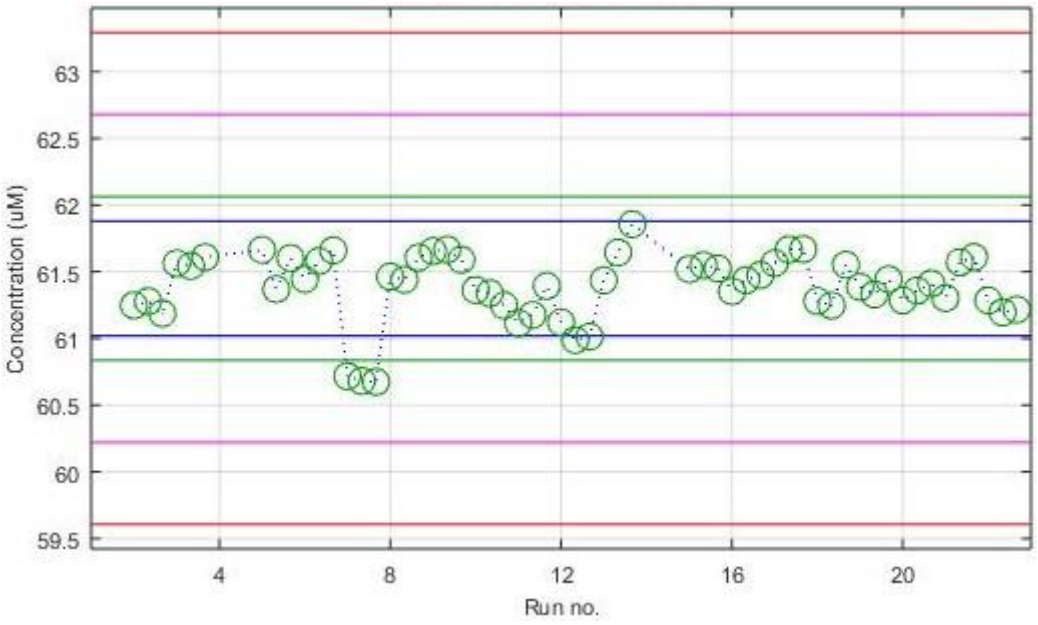
RMNS	NO_3	NO_2	NO_3+ NO_2 (NO_X)	PO_4	SiO_4
lot BW	25.18	0.069	25.25	1.578	61.45
expanded uncertainty	0.20	0.010	0.21	0.014	0.43

KANSO publishes the RMNS nutrient values in µ mol kg^{-1}. These are converted to µ mol l^{-1} at 21°C. Lot BW is not certified for ammonium. NO_x is derived by adding the NO_3 and NO_2 values.

Plot key. The green pink and red lines are the 1%, 2% and 3% contours from the RMNS certified mean value. Exception: nitrite, the contours are at 0.02 µM increments from the certified value. The blue line is the expanded uncertainty of the certified value.

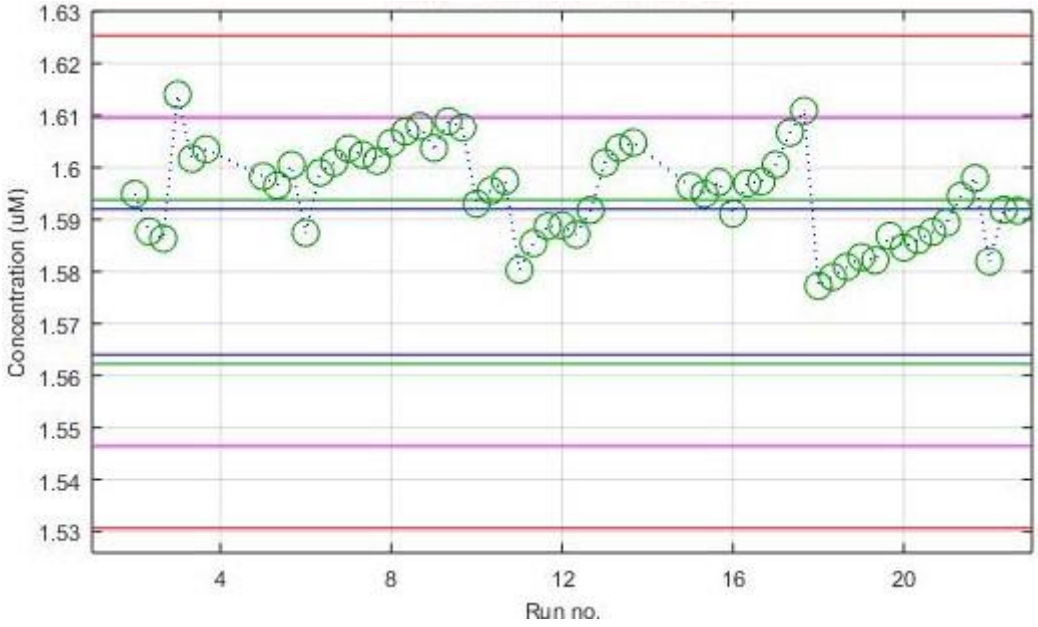
7.5.1 Silicate RMNS Plot

Silicate RMNS (19 runs) for BW (61.45)
Overall mean 61.39 ± 0.25



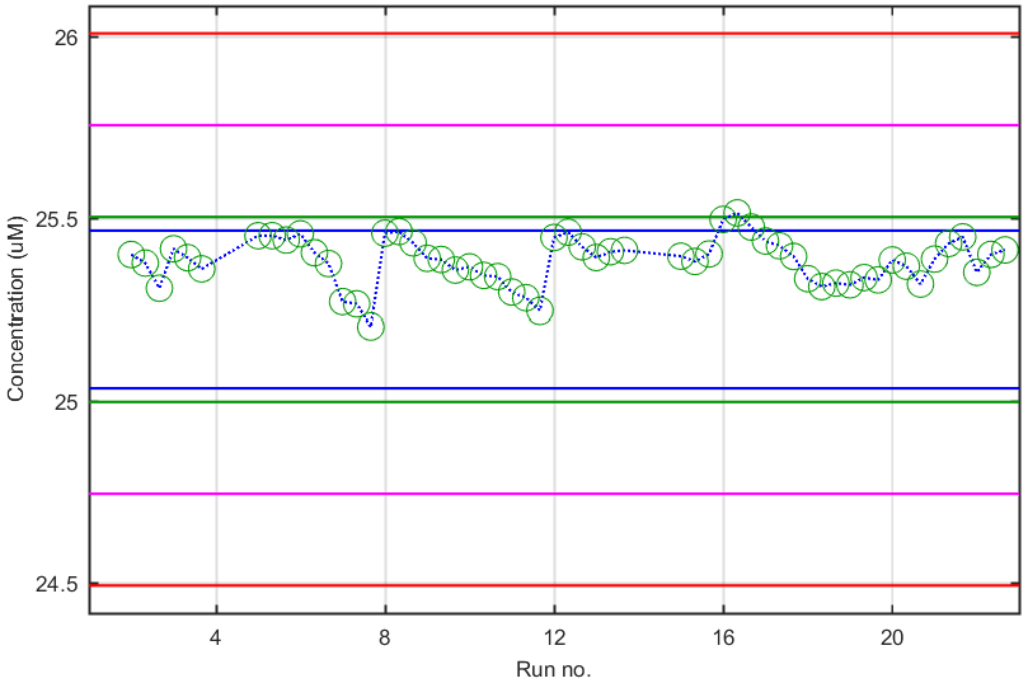
7.5.2 Phosphate RMNS Plot

Phosphate RMNS (19runs) for BW (1.578)
Overall mean 1.591 ± 0.009



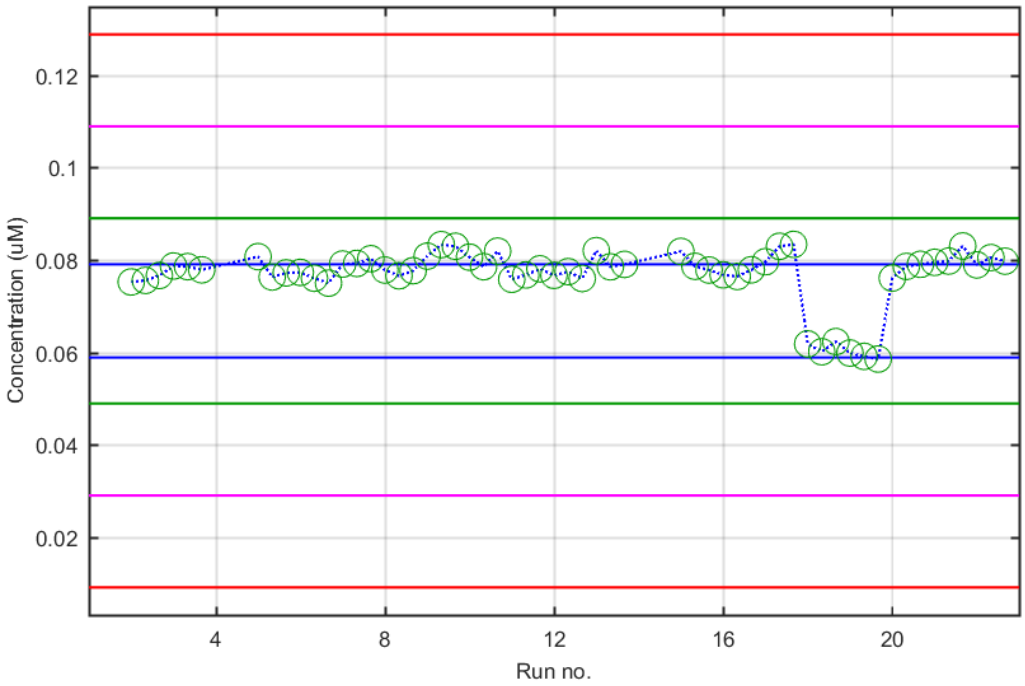
Nitrate + Nitrite (NOx) RMNS Plot

NOx RMNS (19 runs) for BW (25.25)
Overall mean 25.38 ± 0.06



7.5.3 Nitrite RMNS Plot

Nitrite RMNS (19 runs) for BW (0.069)
Overall mean 0.077 ±0.006



7.6 Analytical Precision

The CSIRO Hydrochemistry method measurement uncertainty (MU) has been calculated for each nutrient based on the variation in the calibration curve, calibration standards, pipette and glassware calibration, and precision of the RMNS over time (Armishaw 2003).

	Silicate	Phosphate	Nitrate + Nitrite (NOx)	Nitrite	Ammonia
Calculated MU* @ 1 µmol l^{-1}	±0.017	±0.024	±0.019	±0.14	±0.30[¥]

*The reported uncertainty is an expanded uncertainty using a coverage factor of 2 giving a 95% level of confidence.

[¥]The ammonia MU precision does not include data for the RMNS.

For in2017_v02, the measured detection limits for each run are much lower than the nominal detection limits, indicating high analytical precision at lower concentrations. See appendix 8.3 for the measured MDL per CTD and TMR deployments.

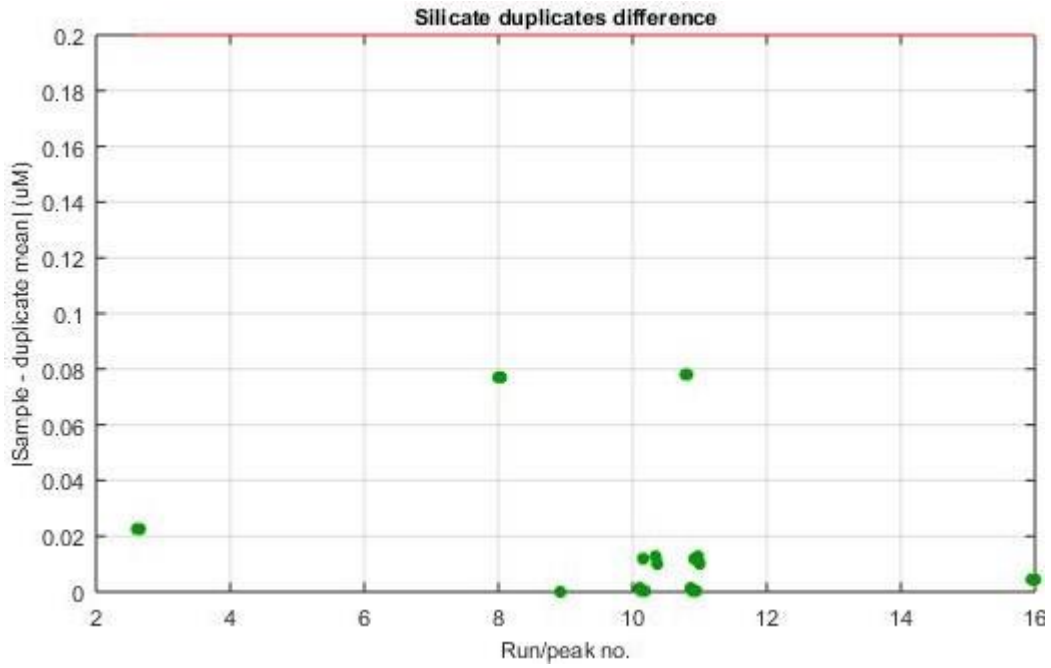
7.7 Sampling Precision

Sampling precision is monitored by assaying duplicate samples collected from the deepest point for each CTD deployment. The sampling precision is good if the difference between the duplicate concentrations is less than the MDL value. The exception is nitrate+nitrite, which uses 0.06 µM as the MDL boundary.

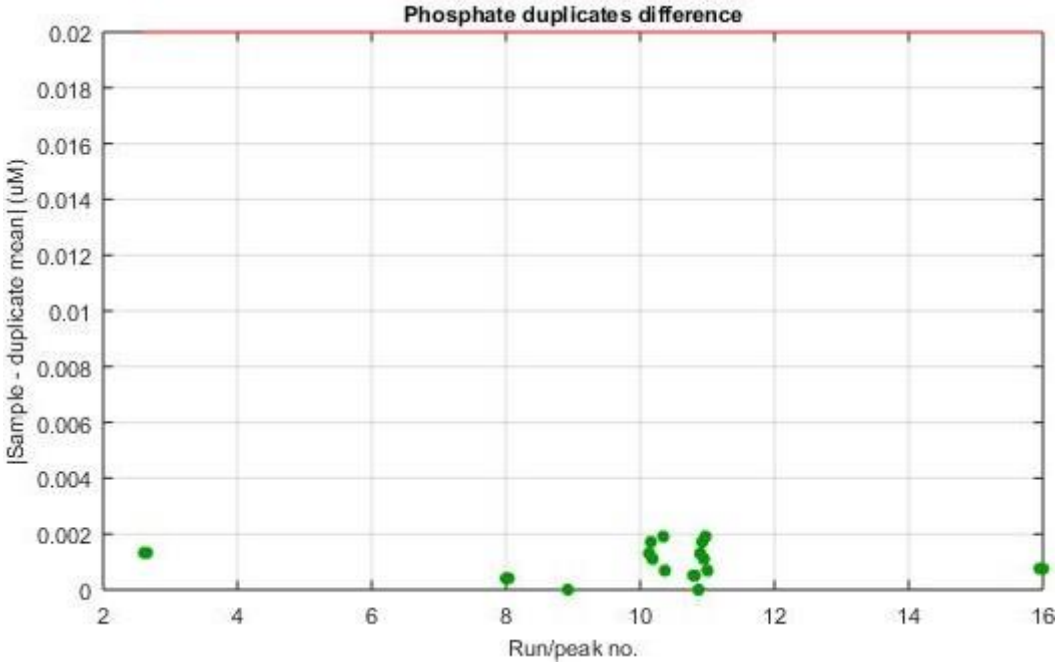
Plots of the difference between the duplicate and their mean for the CTD deployments are below. The red line is the boundary below which sampling precision is deemed good.

For in2018_v02, the sampling precision is good.

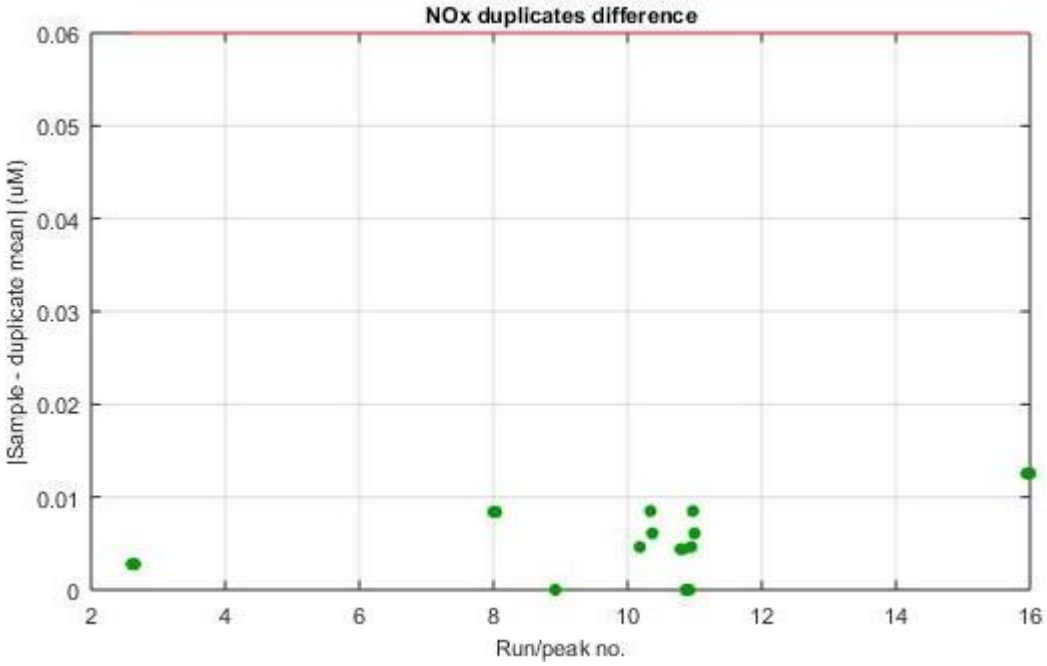
7.7.1 Silicate Duplicates Plot



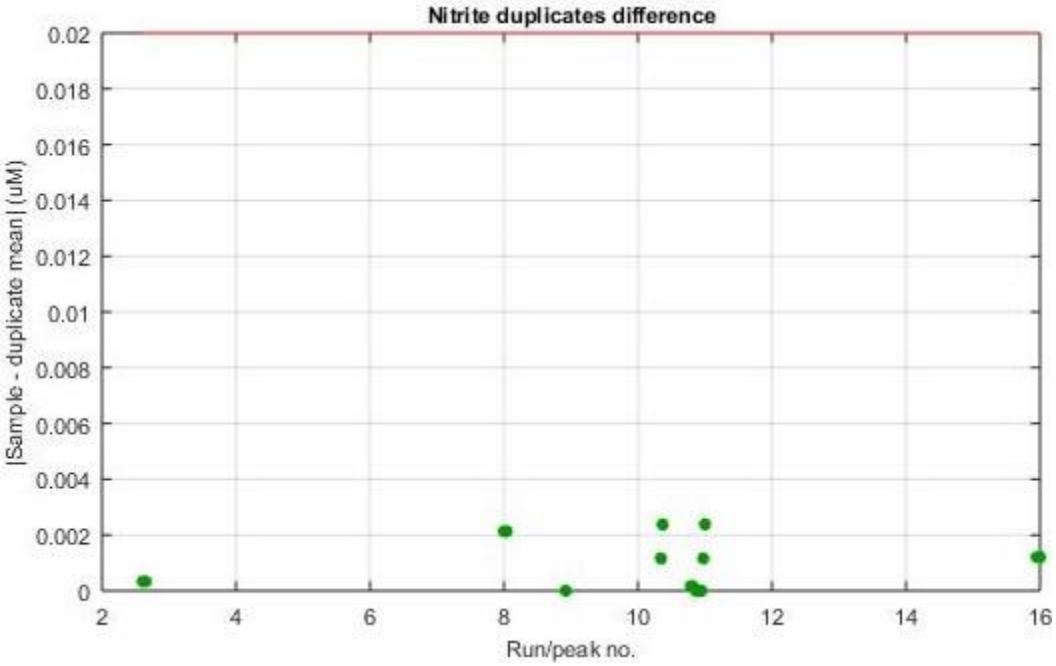
7.7.2 Phosphate Duplicates Plot



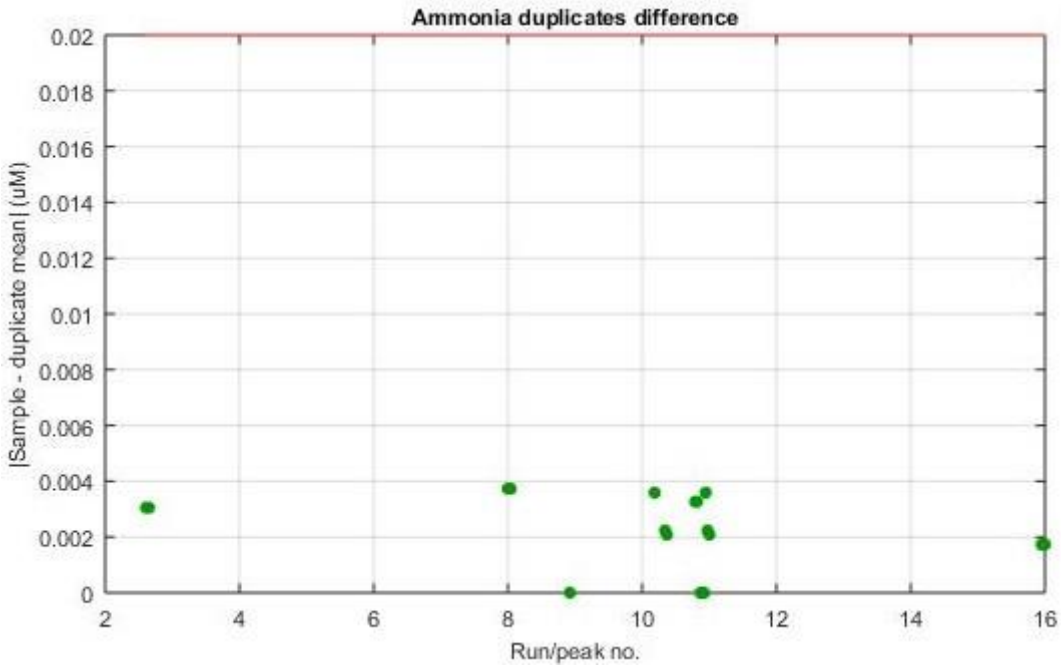
7.7.3 Nitrate + Nitrite (NOx) Duplicates Plot



7.7.4 Nitrite Duplicates Plot

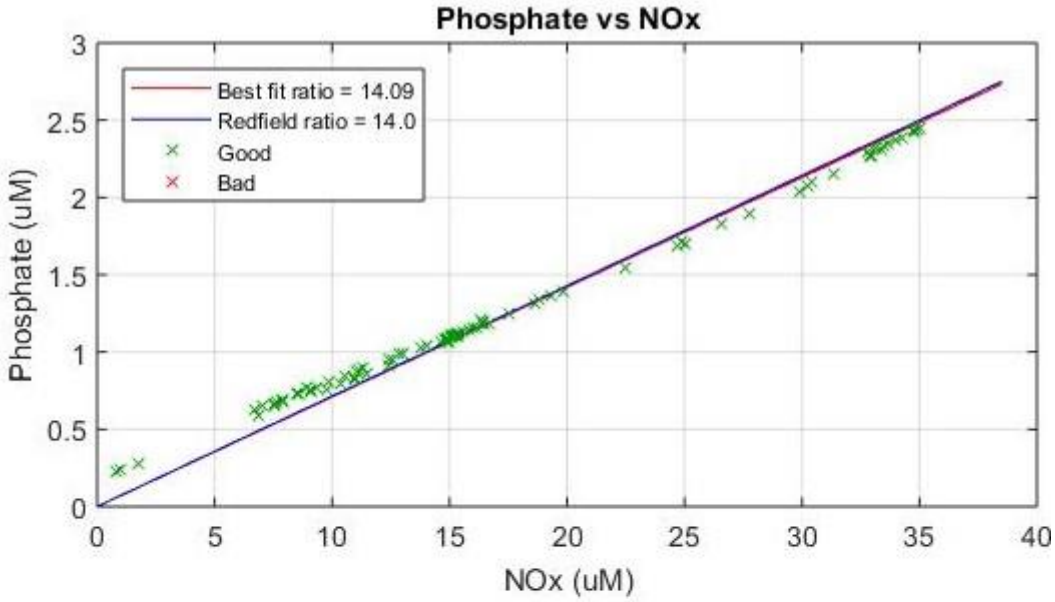


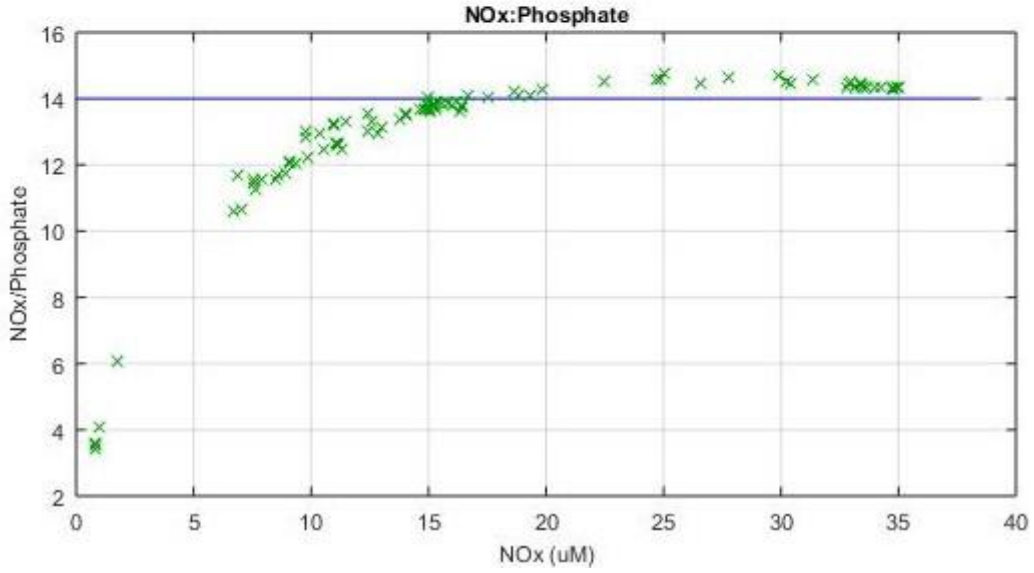
7.7.5 Ammonia Duplicates Plot



7.8 Redfield Ratio Plot (14.0) for CTD Deployments.

Plots consist of phosphate versus NOx for all CTD deployments. Best fit ratio = 14.09





7.9 Missing or Suspect Nutrient Data.

The table below identifies all flagged data and any samples that had repeated analyses performed to obtain good data. Data that falls below the detection limit, Flag 63, is not captured in this table. All GOOD data is flagged 0 in the .csv and .netcdf files. Data that is flagged BAD is not exported to the .csv files. Flag Key in Appendix 8.5.

CTD	RP	Run	Analysis	Flag	Reason for Flag or Action
4	8, 10, 13, 32	Nut009	All	none	No result. Operator error, samples not collected. Unable to collect sample when error realised, Niskin bottles had been emptied. Assayed residue solution in Niskin in run nut009 – unsuitable due to contamination. Results/ samples not included in hydrology data set.

7.10 Temperature & Humidity Change over Nutrient Analyses

Temperature and humidity was not logged during the voyage. The data loggers could not be initiated at the start of the voyage due to an instrument/software fault.

The laboratory temperature was measured and recorded on the nutrient run sheets at the start each analysis run. The temperature varied between 21 and 23°C over the course of the voyage.

8 Appendix

8.1 Salinity Reference Material

OSIL IAPSO Standard Seawater. Batch P161. K_{15} ratio 0.99987. Use by 03/05/2018

8.2 Nutrients: RMNS results for each CTD & TMR Deployment.

Analysis Run	Deployment		RMNS lot BW results (µmole l^{-1})			
	CTD	TMR	SiO_4	PO_4	NO_2	NO_x
BW value			**61.45**	**1.578**	**0.069**	**25.25**
2	2	1	61.24	1.590	0.076	25.36
5	3	2	61.55	1.598	0.078	25.45
6	-	3	61.56	1.596	0.076	25.41
8	4	-	61.50	1.607	0.077	25.45
9	-	4	61.63	1.607	0.082	25.38
10	5	-	61.32	1.595	0.080	25.35
15	6	-	61.53	1.596	0.079	25.39
20	-	5	61.35	1.586	0.078	25.36

8.3 Nutrients: Measured Detection Limit for each CTD & TMR Deployment.

Analysis Run	Deployment		Measured Detection Limit (µmole l^{-1})				
	CTD	TMR	SiO_4	PO_4	NO_2	NO_x	NH_4
2	2	1	0.02	0.015	0.003	0.002	0.003
5	3	2	0.01	0.005	0.004	0.003	0.002
6	-	3	0.12	0.011	0.001	0.003	0.007
8	4	-	0.03	0.002	0.002	0.008	0.004
9	-	4	0.05	0.003	0.005	0.007	0.003
10	5	-	0.04	0.005	0.000	0.004	0.004
15	6	-	0.03	0.008	0.003	0.003	0.003
20	-	5	0.07	0.005	0.005	0.007	0.002

8.4 Nutrients: RMNS results for all AA3HR analysis runs with samples.

Analysis Run	RMNS lot BW results (µmole l^{-1})			
	SiO_4	PO_4	NO_2	NO_x
BW value	**61.45**	**1.578**	**0.069**	**25.25**
2	61.24	1.590	0.076	25.36
5	61.55	1.598	0.078	25.45
6	61.56	1.596	0.076	25.41
7	61.69	1.602	0.079	25.24
8	61.50	1.607	0.077	25.45
9	61.63	1.607	0.082	25.38
10	61.32	1.595	0.080	25.35
11	61.23	1.585	0.077	25.27
12	61.04	1.589	0.077	25.44
13	61.65	1.603	0.080	25.40
15	61.53	1.596	0.079	25.39
16	61.42	1.595	0.077	25.49
17	61.63	1.606	0.082	25.42
18	61.36	1.579	0.062	25.32
19	61.39	1.584	0.059	25.33
20	61.35	1.586	0.078	25.36
21	61.50	1.594	0.081	25.42
22	61.23	1.588	0.080	25.39

8.5 Flag Key for Hydrology Data Set

Flag	Meaning
0	Data is GOOD
63	Below nominal detection limit.
65	Peak shape is suspect.
69	Data flagged suspect by operator.
79	Method Detection Limit (MDL) during run was equal to or greater than nominal MDL. Data flagged as suspect.
129	Peak exceeds maximum detector value. Data is BAD.
133	Error flagged by operator. Data is BAD.
134	Error flagged by software. Peak shape is bad - Median Absolute Deviation (MAD) analysis used. Standards, MDL's and Duplicates deviate from the median, Calibration data falls outside set limits.
141	Missing data, no result for sample ID. Used in the netcdf file. Not used in csv file.
192	Data not processed.

8.6 GO-SHIP Specifications

8.6.1 Salinity

Accuracy of 0.001 is possible with Autosal™ salinometers and concomitant attention to methodology. Accuracy with respect to one particular batch of Standard Sea Water can be achieved at better than 0.001 PSS-78. Autosal precision is better than 0.001 PSS-78. A precision of approximately 0.0002 PSS-78 is possible following the methods of Kawano with great care and experience. Air temperature stability of ± 1°C is very important and should be recorded[2].

8.6.2 Dissolved Oxygen

Target accuracy is that 2 sigma should be less than 0.5% of the highest concentration found in the ocean. Precision or reproducibility (2 sigma) is 0.08% of the highest concentration found in the ocean.

8.6.3 SiO2

Approximately 1-3% accuracy[1], 0.2% precision[3], full scale.

8.6.4 PO4

Approximately 1-2% accuracy[1], 0.4% precision[3], full scale.

8.6.5 NO3

Approximately 1% accuracy[1], 0.2% precision[3], full scale.

8.6.6 Notes

1 If no absolute standards are available then accuracy should be taken to mean the reproducibility presently obtainable in the better laboratories.

2 Keeping constant temperature in the room where salinities are determined greatly increases their quality. Also, room temperature during the salinity measurement should be noted for later interpretation, if queries occur. Additionally, monitoring and recording the bath temperature is also recommended. The frequent use of IAPSO Standard Seawater is endorsed. To avoid the changes that occur in Standard Seawater, the use of the most recent batch is recommended. The bottles should also be used in an interleaving fashion as a consistency check within a batch and between batches.

3 Developments of reference materials for nutrients are underway that will enable improvements in the relative accuracy of measurements and clearer definition of the performance of laboratories when used appropriately and the results are reported with the appropriate meta-data.

9 References

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