

RV INVESTIGATOR

HYDROCHEMISTRY DATA PROCESS REPORT

Voyage:IN2017_v01Chief Scientist:Dr Leanne ArmandVoyage title:Interactions of the Totten Glacier with the Southern Ocean through
multiple glacial cycles

Report compiled by:

Peter Hughes



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1 Itinerary

Depart Leg	Date	Local Time
Hobart	14 January 2017	1800
Arrive	Date	Time
Hobart	5 March 2017	0900

2 Key Personnel

Name	Role	Organisation
Leanne Armand	Chief Scientist	Macquarie University
Doug Thost	Voyage Manager	CSIRO
Peter Hughes	Hydrochemist	CSIRO

3 Summary

Finalized hydrology data can be obtained from the CSIRO data centre. Contact: DataLibrariansOAMNF@csiro.au

3.1 Hydrochemistry

Analysis parameter	Total	Processing Status at voyage end
Nutrients (Seal AA3)	197 CTD	Completed
	52 uwy	
	9 exp	
Salinity (Guildline salinometer)	197 CTD	Completed
	52 uwy	
	9 exp	
Dissolved Oxygen	197 CTD	Completed
	52 uwy	

Note:

- CTD; samples collected from NISKIN bottles on CTD rosette.
- uwy; samples collected from underway clean instrument seawater supply in the PCO2 lab.
- exp; samples collected from the multi-corer niskin and the seawater in the core tubes. Seawater transferred to carboys then sub-sampled for hydrochemistry.

3.2 Rosette and CTD

• 26 stations were sampled with OceanTest Equipment 12l bottles attached to the 24 bottle rosette.

3.3 Data Procedure Summary

After the samples are assayed, their results are processed and collated with ctd deployment metadata by the CSIRO program, HyPro. Final output is in csv and netcdf format. See figure 1.



Figure 1: The processing steps for hydrology data following sample assay.

4 Salinity Data Processing

4.1 Salinity Parameter Summary

Details					
HyPro Version	4.16				
Instrument	Guildline Autosal Laboratory Salinometer 8400(B) – SN 71613				
Software	OSIL Data Logger ver 1.2				
Methods	Hydrochemistry Operations Manual + Quick Reference Manual				
Accuracy	± 0.001 practical salinity units				
Analyst	Peter Hughes				
Lab Temperature	21 -23°C during analysis.				
Bath Temperature	24°C				
Reference Material	Osil IAPSO - Batch P158				
Sampling Containers	200 ml volume OSIL bottles made of type II glass (clear) with disposable plastic insert and plastic screw cap.				
Sample Storage	Samples held in Salt Room for 7-8 hrs to reach ambient temperature before analysis.				
Comments	No issues.				

4.2 Salinity Method

The method uses 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.

The Autosal is calibrated with standard seawater (OSIL, IAPSO) of known conductivity ratio against which the samples are measured. The Autosal is calibrated before each batch run of samples.

Salinity samples are collected into 200ml OSIL bottles –from the bottom via a PTFE straw filled till overflowing. The sample is decanted to allow a headspace of approximately 25cm³. A plastic insert is fitted, the bottle inverted and rinsed then capped and stored cap-down until measured. To measure, the salinometer 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 conductivity ratio data is captured by the Osil data logger v1.2 program which then calculates the practical salinity.



4.3 Unprocessed CTD vs Hydro Salinities Plot

Туре	RP	Analysis	Flag	Reason for Flag or Action
CTD 3	12	Salinity	141 (nc)	No Result. Sample collected but not assayed due to human error. Unable to re-sample as niskin had been emptied.
uwy 27	n/a	Salinity	n/a	No Result. Sample collected but not assayed due to human error.

4.4 Missing or Flagged Salinity Data.

4.5 Salinity Laboratory Temperature.

The salinity laboratory ambient temperature was recorded at two minute intervals using a HOBO temperature logger. The temperature was measured 1.8m above the deck approximately 20cm from the inboard wall of the salinity lab.

The temperature was reasonably stable for the duration of salinity measurements.

Average temperature: 22.0°C, stdev 0.7

5 Dissolved Oxygen Data Processing

5.1 Dissolved Oxygen Parameter Summary

Details					
HyPro Version	4.16				
Instrument	Automated Photometric Oxygen system				
Software	SCRIPPS				
Methods	SCRIPPS				
Accuracy	0.01 ml/l + 0.5%				
Analyst	Peter Hughes				
Lab Temperature (±1°C)	Variable, 21 - 23°C				
Sample Containers	Pre-numbered 140 mL glass iodine determination flasks w/stopper, arranged in boxes excluding light.				
Sample Storage	Samples were stored in the Hydrochemistry lab in their boxes. All samples were analysed within 7 days.				
Comments	No issues.				

5.2 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).

Manganese chloride followed by alkaline iodide, is added to the sample, and the precipitated manganous hydroxide is distributed evenly throughout the bottle by shaking. At this stage, the dissolved oxygen oxidizes an equivalent amount of Mn(II) to Mn(IV). Just before titration, the sample

is acidified, converting the Mn (IV) back to the divalent state liberating an amount of lodine equivalent to the original dissolved oxygen content of the water. The lodine is auto-titrated with a standardised thiosulphate solution using a Metrohm 665 Dosimat 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.

The thiosulphate solution is standardised by titrating a 10ml aliquot of potassium iodate primary standard. The blank correction is determined from the difference between two consecutive titres for 1ml aliquots of the same potassium iodate solution.



5.3 Unprocessed CTD vs Hydro DO Plot



5.4 Dissolved Oxygen Thiosulphate Normality across voyage

5.5 Dissolved Oxygen Blank Titre across voyage



5.6 Missing or Flagged Dissolved Oxygen Data.

No missing or flagged data.

6 Nutrient Data Processing

6.1 Nutrient Parameter Summary

Details						
HyPro Version	4.16					
Instrument	Seal AA3 HR					
Instrument Software	Seal AACE 6.10					
Methods	AA3 Analysis M	ethods internal i	manual			
Nutrients analysed	Silicate Phosphate Nitrate + Nitrite Amm					
Concentration range	140 µmol l ⁻¹	3 µmol l-1	42 μmol l ⁻¹	1.4 µmol l⁻¹	2.0 μmol l ⁻¹	
Method Detection Limit (MDL)	0.2 μmol l ⁻¹	0.02 µmol l ⁻¹	0.02 µmol l ⁻¹	0.02 µmol l ⁻¹	0.02 µmol l ⁻¹	
Analyst	Peter Hughes					
Lab Temperature (±1°C)	Variable, 21 – 2	23°C				
Reference Material	Kanso RMNS lo	t BW, CA and CC				
Sampling Container type	50 ml HDPE bot	ttles				
Sample Storage	CTD: < 4 hrs at room temperature or ≤ 24 hrs @ 4°C					
	Uwy & Exp: frozen at -20C, thawed at room temperature prior to assay.					
Pre-processing of Samples	None					
Comments	All data recorde	ed; no issues enc	ountered during	voyage.		

6.2 Nutrient Methods

CSIRO Oceans and Atmosphere Hydrochemistry nutrient analysis is performed with a segmented flow auto-analyser – Seal AA3 HR – to measure silicate, phosphate, nitrite, nitrate plus nitrite (NOx), and ammonium

Silicate: 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: 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 molybate. It is then reduced by ascorbic acid and its absorbance is measured at 880nm.

Nitrate: colourimetric analysis, Cu-Cd reduction – Naphthylenediamine photometric 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 dihydrochloride to produce a reddish purple azo complex and its absorbance is measured at 520 nm.

Nitrite: colourimetric analysis, Naphthylenediamine photometric method. As per nitrate method without the copper cadmium reduction column and buffer.

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

Detailed SOPs can be obtained from the CSIRO Oceans and Atmosphere Hydrochemistry Group on request.

6.3 Instrument Calibration and Data Parameter Summary

All instrument parameters and reagent batch compositions are logged for each analysis run. This information is available on request.

The raw data from each analysis run on the Seal AA3HR is imported into HyPro for peak height determination, constructing the calibration curve, deriving the sample results and applying drift and carry-over corrections. The calibration curve is fitted to the standards by performing several passes over each standard point and weighting its contribution to the curve depending on the magnitude of the difference between its measured and calculated value. The larger the difference, the less weighting is given to the standard's contribution towards the curve construction. 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).
- 0.02uM for phosphate, nitrite and ammonium.

Following standard procedures, the operator may choose to not include bad calibration points (see section 6.6 for edited data). Below are the corrections and settings that Hypro applied to the raw data.

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	Quadractic	
Forced through zero?	N	N	N	N	Ν	
# of points in Calibration	6	5	6	5	5	
Matrix Correction	N	N	N	N	Ν	
Blank Correction	N	N	Ν	Ν	Ν	
Carryover Correction (Hypro)	Y	Y	Y	Y	Y	
Baseline Correction (Hypro)	Y	Y	Y	Y	Y	
Drift Correction (Hypro)	Y	Y	Y	Y	Y	
Data Adjusted for RMNS	N	N	Ν	Ν	Ν	
Peak Window Defined	HyPro	HyPro	HyPro	HyPro	HyPro	
Medium of Standards	LNSW (bulk on through a 10 n laboratory at 2	NSW (bulk on deck of Investigator) collected on 28/9/2016. Sub-lot passed nrough a 10 micron filter and stored in 20 L carboys in the hydrochemistry aboratory at 22°C.				
Medium of Baseline	18.2 Ω H ₂ O					
Proportion of samples in duplicate	1 duplicate for each CTD deployment from the deepest point. Usually rosette position 1.					
Comments	Calibration and QC data that was edited or removed is located in the table in section 6.7. The reported data is not corrected to the RMNS. Deployment vs RMNS data in Appendix 7.3					

6.4 Accuracy - Reference Material for Nutrients in Seawater (RMNS) Plots

Japanese KANSO certified reference materials (RMNS) for silicate, phosphate, nitrate and nitrite in seawater was used in each nutrient analysis run to determine the accuracy. For each analysis run, a new RMNS bottle was opened and used. The RMNS was assayed in quadruplicate after the calibration standards.

RMNS lots BW, CA and CC were used. Their stated values in μ mol/kg are converted to μ mol l⁻¹ at 21°C and are listed below. RMNS do not have certified ammonium values.

RMNS	NO ₃	NOx	NO ₂	PO ₄	SiO ₄
BW	25.18 ± 0.20	25.25 ± 0.21	0.069 ± 0.010	1.578 ± 0.014	61.45 ± 0.42
СА	20.13 ± 0.15	20.20 ± 0.16	0.065 ± 0.010	1.441 ± 0.014	37.46 ± 0.23
СС	31.62 ± 0.24	31.74 ± 0.24	0.119 ± 0.005	2.130 ± 0.019	88.23 ± 0.49

Table 1: RMNS BW, CA and CC concentrations (μ M) at 21°C

The submitted nutrient results do NOT have RMNS corrections applied.

The following plots show RMNS values within 1% (green lines), 2% (pink lines) and 3% (red lines) of the published RMNS value except for nitrite. The nitrite limit is set to $\pm 0.02 \ \mu$ M (MDL) as 1% is below the method MDL. The GO-SHIP criteria (Hyde *et al.*, 2010), specifies using 1-3 % of full scale (depending on the nutrient) as acceptable limits of accuracy. The assayed RMNS values per CTD deployment are reported in Appendix 7.3.



6.4.1 Silicate RMNS Plot









6.4.2 Phosphate RMNS Plot









6 Run no.

6.4.3 Nitrate + Nitrite (NOx) RMNS Plot

31



Nitrite RMNS (1 run) for CA(0.065) Overall mean 0.086 +- 0.0008





6.4.4 Nitrite RMNS Plot

6.5 Analytical Precision

The CSIRO Hydrochemistry method measurement uncertainty (MU) has been calculated for each nutrient based on 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
Calculated MU* @ 1 μ mol l ⁻¹	±0.017	±0.020	±0.017	±0.108

*expanded uncertainty using a coverage factor of 2 giving a 95% level of confidence.

The measured method detection limits (MDL) achieved during the voyage are lower than the nominal detection limits, indicating high precision at lower concentrations. RMNS and MDL precision data listed below. Units are μ mol l⁻¹.

	Silicate	Phosphate	Nitrate + Nitrite (NOx)	Nitrite	Ammonium
Nominal MDL	0.20	0.02	0.02	0.02	0.02
Min	0.01	0.003	0.002	0.001	0.002
Max	0.11	0.019	0.007	0.005	0.017
Mean	0.04	0.009	0.004	0.003	0.007
Median	0.03	0.008	0.004	0.003	0.007
Precision of MDL (stdev)	0.03	0.004	0.002	0.001	0.004
Published RMNS BW	61.45	1.578	25.249	0.069	n/a
w/uncertainty	± 0.42	± 0.014	± 0.21	± 0.010	
RMNS Min	61.11	1.568	25.25	0.080	-
RMNS Max	62.01	1.616	25.65	0.084	-
RMNS Mean	61.52	1.588	25.48	0.082	-
RMNS Median	61.49	1.584	25.48	0.082	-
RMNS Std Dev	0.26	0.013	0.09	0.001	-

6.6 Sampling Precision

Duplicates of the deepest sample were assayed to monitor overall precision. The precision is deemed good if the difference between duplicate results and their mean is below the MDL for silicate, phosphate and nitrite and within 0.05 μ M for nitrate. Duplicate plots below.



6.6.1 Silicate Duplicate Plot



6.6.2 Phosphate Duplicate Plot



6.6.3 Nitrate + Nitrite (NOx) Duplicate Plot



6.6.4 Ammonia Duplicate Plot



6.6.5 Nitrite Duplicate Plot

6.6.6 Redfield Ratio Plot (14.0)

Plots consists of phosphate versus NOx, best fit ratio = 14.56





6.7 Edited Calibration Data

All calibration data was left as is for Hypro to evaluate. All calibrations were deemed 'Good'. Further details on per run quality of the calibration can be provided on request.

6.8 Missing or Flagged Nutrient Data

The table below identifies all flagged data and missing results. 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 .nc files. Refer to Appendix 7.2 for flag explanations.

CTD	RP	Analysis	Flag	Reason for Flag or Action
4	1	Nutrients NOx	0	Duplicates outside of MDL cut-off (0.02uM). Initially flagged 69 by HyPro, manually changed to 0 as they are within 0.05uM of their mean. Duplicate results (uM): 33.25, 33.31
10	1	Nutrients NOx	0	Duplicates outside of MDL cut-off (0.02uM). Initially flagged 69 by HyPro, manually changed to 0 as they are within 0.05uM of their mean. Duplicate results (uM): 32.89, 32.80
17	1	Nutrients NOx	0	Duplicates outside of MDL cut-off (0.02uM). Initially flagged 69 by HyPro, manually changed to 0 as they are within 0.05uM of their mean. Duplicate results (uM): 32.81, 32.86
28	1	Nutrients NOx	0	Duplicates outside of MDL cut-off (0.02uM). Initially flagged 69 by HyPro, manually changed to 0 as they are within 0.05uM of their mean. Duplicate results (uM): 32.51, 32.61
Uwy / exp	Sample #	Analysis	Flag	Reason for Flag or Action
uwy	34	Nutrients	141	No results. Sample collected. Not assayed due to human

6.9 Hydrology Laboratory Temperature.

The laboratory temperature was recorded at two minute intervals using a HOBO temperature logger. The temperature was measured 40cm above the main chemistry module of the Seal AA3HR instrument.

error.

The temperature was reasonably stable for the duration of nutrient analysis. Data available on request.

Average temperature 22.4°C, stdev 0.7

7 Appendix

7.1 Salinity Reference Material

Reference	Batch	Use by Date	K 15
Osil IAPSO Standard Seawater	P158	25/03/2018	0.99940

7.2 Reference Material Nutrient Seawater, KANSO Japan

RMNS	NO _x	NO ₃	NO ₂	PO ₄	SiO ₂
BW	25.25 ± 0.21	25.18 ± 0.20	0.069 ± 0.010	1.578 ± 0.014	61.45 ± 0.42
СА	20.20 ± 0.16	20.13 ± 0.15	0.065 ± 0.010	1.441 ± 0.014	37.46 ± 0.23
СС	31.74 ± 0.24	31.62 ± 0.24	0.119 ± 0.005	2.130 ± 0.019	88.23 ± 0.49

7.3 RMNS results for each CTD deployment

CTD	SiO ₄	SiO ₄	PO ₄	PO ₄	NO ₂	NO ₂	NOx	NOx
	measured	expected	measured	expected	measured	expected	measured	expected
1	61.5	61.5	1.58	1.58	0.083	0.069	25.47	25.25
2	62.0	61.5	1.60	1.58	0.082	0.069	25.53	25.25
3,4	37.7	37.5	1.45	1.44	0.086	0.065	20.38	20.20
5	61.9	61.5	1.58	1.58	0.084	0.069	25.65	25.25
6	62.0	61.5	1.59	1.58	0.084	0.069	25.58	20.20
7,8	88.6	88.2	2.16	2.13	0.133	0.119	32.08	31.74
9	61.5	61.5	1.58	1.58	0.081	0.069	25.36	25.25
10	61.6	61.5	1.62	1.58	0.084	0.069	25.39	25.25
16,17	61.6	61.5	1.57	1.58	0.081	0.069	25.50	25.25
19	61.5	61.5	1.57	1.58	0.083	0.069	25.54	25.25
20,21	61.8	61.5	1.58	1.58	0.082	0.069	25.56	25.25
22	61.3	61.5	1.58	1.58	0.082	0.069	25.49	25.25
23	61.3	61.5	1.58	1.58	0.080	0.069	25.45	25.25
25,26	61.1	61.5	1.60	1.58	0.082	0.069	25.45	25.25
27,28,29	61.5	61.5	1.61	1.58	0.082	0.069	25.42	25.25
30	61.3	61.5	1.59	1.58	0.082	0.069	25.39	25.25
31,32	61.2	61.5	1.58	1.58	0.083	0.069	25.48	25.25
33	61.3	61.5	1.60	1.58	0.082	0.069	25.48	25.25

7.4 Hypro Flag Key for CSV & NC file

Flag	Meaning
0	Data is GOOD.
63	Result below nominal detection limit.
69	Duplicate difference from their mean is outside the MDL cut-off limit.
141	No result. Flag used in the .nc file. Flag not used in the .csv file.
192	Data is unprocessed.

CTD	RP	Analysis	Flag	Reason for Flag or Action
3	12	Salinity	141	No Result. Sample collected. Not assayed due to human error.
4	1	Nutrients NOx	0	Duplicates outside of MDL cut-off (0.02uM). Initially flagged 69 by HyPro, manually changed to 0 as they are within 0.05uM of their mean. Duplicate results (uM): 33.25, 33.31
10	1	Nutrients NOx	0	Duplicates outside of MDL cut-off (0.02uM). Initially flagged 69 by HyPro, manually changed to 0 as they are within 0.05uM of their mean. Duplicate results (uM): 32.89, 32.80
17	1	Nutrients NOx	0	Duplicates outside of MDL cut-off (0.02uM). Initially flagged 69 by HyPro, manually changed to 0 as they are within 0.05uM of their mean. Duplicate results (uM): 32.81, 32.86
28	1	Nutrients NOx	0	Duplicates outside of MDL cut-off (0.02uM). Initially flagged 69 by HyPro, manually changed to 0 as they are within 0.05uM of their mean. Duplicate results (uM): 32.51, 32.61

7.5 All Flagged and Missing Data

Uwy / exp	Sample #	Analysis	Flag	Reason for Flag or Action
uwy	27	Salinity	141	No result. Sample collected. Not assayed due to human error.
uwy	34	Nutrients	141	No results. Sample collected. Not assayed due to human error.

7.6 GO-SHIP Specifications

- Salinity Accuracy of 0.001 is possible with Autosal[™] salinometers and concomitant attention to methodology, e.g., monitoring Standard Sea Water. 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. High precision of approximately 0.0002 PSS-78 is possible following the methods of Kawano (this manual) with great care and experience. Air temperature stability of ± 1°C is very important and should be recorded.¹
- O₂ 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.
- SiO_2 Approximately 1-3% accuracy^{†,2}, and 0.2% precision, full-scale.
- PO₄ Approximately 1-2% accuracy^{\dagger ,2}, and 0.4% precision, full scale.
- NO₃ Approximately 1% accuracy^{†,2}, and 0.2% precision, full scale.
- Notes: [†]If no absolute standards are available for a measurement then accuracy should be taken to mean the reproducibility presently obtainable in the better laboratories.

¹ 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 batches is recommended. The bottles should also be used in an interleaving fashion as a consistency check within a batch and between batches.

² 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

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