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Southern Ocean Time Series (SOTS) Quality Assessment and Control Report Remote Access Sampler: Sample Analysis Version 1.0

Macronutrient analysis

2009-2018

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Citation

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Important disclaimer

Foreword

The Southern Ocean Time Series (SOTS) is a Sub-Facility of the Australian Integrated Marine Observing System (IMOS), funded by the National Collaborative Research Infrastructure Strategy (NCRIS). It is operated under collaborative arrangements among the CSIRO Oceans and Atmosphere, Bureau of Meteorology, and University of Tasmania, including via the Antarctic Climate and Ecosystems Cooperative Research Centre and the Australian Antarctic Program Partnership. The primary focus is sustained observing of ocean properties and processes important to climate, carbon cycling, and ocean productivity.

The SOTS Sub-Facility consists of deep ocean moorings deployed in Subantarctic waters southwest of Tasmania, equipped with autonomous sensors and sample collectors. SOTS moorings are serviced annually - the existing moorings are recovered and new moorings are deployed. Some sensor data is transmitted from the moorings via satellite in near real time. Other sensor data and samples are recovered during the annual service visit.

This report details the quality assessment and control procedures applied to the macronutrient samples from the Remote Access Sampler deployed on SOFS and Pulse moorings. The datasets are publicly available via the AODN Portal https://portal.aodn.org.au/search.

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Acknowledgments

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SOTS is a member of the OceanSITES global network of time series observatories. (www.OceanSITES.org).

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Executive summary

The Southern Ocean Time Series (SOTS) Observatory located near 140°E and 47°S provides high temporal resolution observations in sub-Antarctic waters. It is focused on the sub-Antarctic Zone because waters formed at the surface in this region slide under warmer subtropical and tropical waters, carrying CO_2 and heat into the deep ocean, where it is out of contact with the atmosphere. This process also supplies oxygen for deep ocean ecosystems, and exports nutrients that fuel ~70% of global ocean primary production. This region is also the boundary between the nutrient rich waters of the Southern Ocean and the oligotrophic subtropical gyres to the north. These processes are sensitive to climate change, but the probable nature and impacts are not yet known.

This report details the quality control procedures applied to the data from samples collected by the Remote Access water Sampler (McLane RAS 500) deployed on the SOTS and Pulse moorings between 2009 and 2019. The quality-controlled datasets are publicly available via the IMOS Data Portal. This report should be consulted when using the data.

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1 Introduction

Detailed descriptions of mooring designs, locations and sample collections are provided in the SOTS Annual Reports, which are divided into three parts,

Report 1. Overview, listing mooring voyages, dates, locations, designs and instruments;

Report 2. Samples, detailing the sample collections and

Report 3. Sensors, which contains descriptions and data QC procedures of the sensors mounted on the moorings.

The reports are available at:

https://catalogue-imos.aodn.org.au/geonetwork/srv/eng/metadata.show?uuid=afc166ce-6b34-44d9-b64c-8bb10fd43a07

The RAS sampler deployments are summarized in Table 1 (below). FluxPulse did not return samples because of mooring failure and loss of the sampler, SOFS-7 failed early on deployment and two samples were collected while adrift and Pulse-9 and Pulse-10 were recovered before the sampler program was completed.

Table 1. Instrument deployment details

Site	Mooring	Lat Dec deg	Long Dec deg	Deployed	Recovered	Depth m	Serial no.	Samples Returned
PULSE	Pulse-6 2009	-46.3224	140.6776	2009-09-28	2010-03-18	32	11906-01	48
PULSE	Pulse-7 2010	-46.9347	142.2583	2010-09-12	2011-04-17	31	11906-01	48
PULSE	Pulse-8 2011	-46.9295	142.2147	2011-08-03	2012-07-19	34	11906-01	48
PULSE	Pulse-9 2012	-46.8493	142.3986	2012-07-17	2013-05-05	38	12709-01	32
PULSE	Pulse-10 2013	-46.9378	142.2847	2013-05-07	2013-10-13	28	11906-01	22
PULSE	Pulse-11 2015	-46.9405	142.3262	2015-03-25	2016-03-19	28	11906-01	48
SOFS	FluxPulse-1 2016	-46.7240	141.9297	2016-03-16	2016-06-23	30	12709-01	0
SOFS	SOFS-7 2018	-47.0111	142.2135	2018-03-06	2018-03-16	4	14384-01	2
SOFS	SOFS-7.5 2018	-47.0227	142.2334	2018-08-22	2019-03-22	4	14384-01	48

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This QC report outlines the tests performed on RAS (McLane RAS 500) samples, and the associated allocation of quality control flags, for macronutrient concentration analyses.

The QC flags are provided in the on-line netcdf files and follow the Argo Table 2A (IMOS, 2015) Flag convention (equivalent to IMOS Standard Flag, OCEANSITES, and IODE flag conventions) and are as follows:

Table 2. QC flag meanings

N	MEANING
0	no QC was performed
1	good data
2	probably good
3	bad data that are potentially correctable
4	bad data
5	value changed
6	not used
7	not used
8	interpolated value
9	missing value

Quality assurance is via careful preparation and deployment of the RAS and handling of the recovered samples. It does not lead to an uncertainty estimate or a flag but is important to understand the overall fidelity of the observations.

Quality Control is via:

1. Quality assessment of the recovered RAS and samples

   This includes the amount collected, data from associated sensors, appearance, etc. and leads to overall sample quality flags.

2. Quality Control procedures for the nutrient analyses

   This includes calibrations, replicates, reference materials (where available), and comparison to nutrient data from CTD bottle casts and other sources that are adjacent spatially and temporally to the SOTS and Pulse moorings. Analytical errors and flags are determined for each analyte.

The reported variables use standard names, or long names if no standard name is available, as follows:

time_of_sample_start (yyyy:mm:dd hh:mm:ss UTC)
depth_actual (m)
depth_nominal (m)
sea_water_temperature (^{o}C)
sea_water_practical_salinity
sample_number (position)
sample_mass (kg)
moles_of_nitrate_and_nitrite_per_unit_mass_in_sea_water (μmol.kg^{-1})
moles_of_phosphate_per_unit_mass_in_sea_water (μmol.kg^{-1})
moles_of_silicate_per_unit_mass_in_sea_water (μmol.kg^{-1})
moles_of_alkalinity_per_unit_mass_in_sea_water (μmol.kg^{-1})
moles_of_inorganic_carbon_per_unit_mass_in_sea_water (μmol.kg^{-1})

2 Quality assurance of sample collection - Preparation of the RAS.

Over the lifetime of the project, the configuration of the sampler has undergone change along with the accompanying sensors. The various configurations and deployments are outlined in the relevant SOTS Annual Reports (Report 2. Samples). Note that the sampler collects whole water samples that are unfiltered, except for Pulse-6, which had inline filters before the sample bags in the RAS, but without isolation of the filter from the sample. The most significant difference is between the Pulse moorings on which the RAS was deployed at 30m depth, and the SOFS mooring on which the RAS was inside the SOFS mooring surface float with an intake at 4.5m depth.

Three samplers are rotated on the moorings, as identified with the following serial numbers and names (see also Table 1). Artemis has had a controller upgrade to allow longer flushing times. Apollo was lost with the Flux-Pulse mooring and Orpheus is the most contemporary.

RAS3-48-500 11906-01 Artemis

RAS3-48-500 12709-01 Apollo

RAS3-48-500 14384-01 Orpheus

The annual SOTS Report 2. Samples does not exhaustively outline the preparation of the instruments from a quality assurance perspective and additional considerations are as follows. Contamination is minimised for all surfaces of the RAS that are in contact with the sample by cleaning with zero phosphate rinseable detergent (2% Neutracon) followed by copious rinsing with milliQ water of all 49 ports via a program that repeatedly steps through the port positions. The quality of the Tedlar sample bags has decreased over the life of the program and they now required increased cleaning and are rinsed at least 3 times with milliQ water to remove fibres, and inspected for failure points. The prime volume milliQ water which displaces all air in the fluid path is boiled to degas it before the priming process. The RAS has collected pairs of samples preserved consecutively with 1% glutaraldehyde (IUPAC pentanedial) and then 80µM mercuric chloride. Because of the possibility of cross-contamination, the distribution valve is parked in the home position once the priming is completed and not driven during poisoning. This has become less important with a revised operation of the RAS in which all samples are now preserved with mercuric chloride.

A test program is run prior to the final poisoning, battery installation and programming to ensure as far a possible that all aspects of the instrument will function when deployed. The sample log is downloaded after recovery and is retained with the raw data and referred to in allocating sample timing, sample numbers, valve flushing and pump operation.

3 Quality assessment of the recovered RAS samples

Sensors included in the RAS package provide data used in the calculation of the reported variables (see the SOTS Annual Reports, Report 3. Sensors).

time_of_sample_start (yyyy:mm:dd hh:mm:ss UTC)

To ensure accurate time stamps for comparison with other sensors, the time is set to UTC against the World Clock. The RAS clock is checked on recovery and has been found to be within 0.5 minutes. Currently no flag is set for the time stamp.

Depth_actual (dbar)

Where possible the actual depth of deployment is obtained, typically from a SeaBird SBE16 or equivalent pressure sensor. In the event of sensor failure, the depth may be the measured length of the mooring. In some cases, interpolation is necessary with a combination of measured length and sensors at other positions. Error in the actual depth is calculated as less than 1m for the Pulse mooring and is variable during the period of deployment due to the use of a flexible element (90m bungee). The Depth_actual is currently not flagged.

Depth_nominal (m)

The nominal depth is the designed target depth and is not flagged.

For deployments of the RAS inside the SOFS surface float, only the nominal depth is reported. The measured length of intake along the mooring chain and the height of the RAS in relation to the sea level scum line on the float is within ±0.2m of the actual depth.

sea_water_temperature (^{o}C)
Temperature is reported with the nutrient data because of the requirement for seawater density to convert concentration units to µmol.kg^{-1}. The CTD sensor included in the RAS package is typically a Seabird16plusV2 with a manufacturer's specification of ±0.005 ^{o}C. Temperature uncertainty (systematic error) is estimated using RMS difference for 1hour gridded data (for more detail refer to the SOTS Annual Reports, Report 3. Sensors). In the event of sensor failure, sensors closest in depth on the mooring are used in the density calculation (provided their data is consistent with any available from the failed sensor), and the temperature is flagged as interpolated (flag 8). The RAS has a temperature sensor included in the controller case but it has low resolution of ±0.1 ^{o}C. It is not used when other data are available, but provides an additional point of reference.

sea_water_practical_salinity (psu)
Salinity is reported with the nutrient data because of the requirement for seawater density to convert concentration units to µmol.kg^{-1}. The CTD sensor included in the RAS package, generally a Seabird16plusV2 (SBE CTD) has a manufacturers specification of ±0.00054 psu and is used in the density calculation and prime volume calculation (refer to the SOTS Annual Reports, Report 3. Sensors for more detail). In the event of sensor failure, sensors closest in depth on the mooring are used in the density calculation (provided their data is consistent with any available from the failed sensor), and the salinity is flagged as interpolated (flag 8), along with an uncertainty (systematic error) estimated using 1-hour gridded data RMS difference between the RAS sensor and the closest sensor.

sample_number (position)
The sample number flag is allocated based on inspection after recovery and the RAS sampler log.

- If the outer acrylic sample tube is intact and the log shows a complete program for the sample it is flagged 1.
- Failure to sample due to an incomplete program is flagged 9, e.g., early mooring recovery.
- A partial sample due to a broken outer acrylic tube is flagged 2 to indicate that the sample is good but has under collected with a reduced sample volume.
- Where damage has resulted in an insufficient sample volume for analysis it is flagged 9.

Considerable care to affix sample numbers is required when unloading because the positions are lost when the samples are removed from the sampler.

sample_mass (kg)

The "pumped volume" extracted from the instrument log on recovery has been found to be inaccurate as it is based on the pump run time, not the volume delivered. Recovered samples are weighed to assess the actual quantity collected (and this is applied to the phytoplankton abundance reported elsewhere - see the SOTS Annual Reports, Report 2. Samples section B.4.4). The quantity pumped can also indicate problems with the sampler. All sample bags are capped after removing them from the acrylic tube. The outside of the bag is wiped dry and the bags weighed to 0.01g and a mean empty weight plus prime volume (n=10, 10.17g ±0.06) is subtracted to give a sample mass.

- If no sample has been lost by the operator during unloading, the sample weight is flagged 1.
- If the loss is small or estimable it is flagged 2.
- If the loss is large (>100mL) it is flagged 4
- if the sample is dropped, or another catastrophic loss occurs, it is flagged 9.

prime volume

Air is displaced from all associated plumbing by priming with boiled milliQ water (>18Mohm). Air bubbles in the delivery tube and pump can cause the sampler to fail due to pinched tubing at depth, interrupted pumping, and insufficient displacement of the water surrounding the Tedlar bag leading to under-collection. Excess prime volume is removed by forward pumping with the onboard pump leaving approximately 5mL of milliQ in the Tedlar sample bags and delivery tubes. The sample bags are then poisoned by injecting 0.25mL saturated mercuric chloride through the compression fittings at the acrylic tube cap. The sample dilution from priming affects all calculated concentrations. It is consistently about 1% of the collected volume but can be more if there are problems with pumping, or with timing when driving the sample distribution valve. The prime volume is calculated as the ratio of the measured salinity obtained during analysis of TCO_2 (using the SBE CTD in the SOMMA) and the salinity as measured by the SBE CTD sensor in the RAS instrument package. The effect on the final nutrient concentrations is small under normal circumstances and does not increase the total sample uncertainty beyond the reported 95% confidence interval and it is currently not flagged. Where sensor data are unavailable, and the sample mass is as expected and has a flag of 1 or 2, a 1% dilution is attributed and flag 2 and comment is added to the metadata. However, it is possible despite all care, that a large dilution factor could arise accidentally during priming and become apparent only when the SOMMA salinity and final nutrient concentrations are considered and may then be flagged 3.

Sample handling

Decanting of samples is described in detail in the SOTS Annual Reports, Report 2. Samples section B.4.3. Briefly, pairs of 10mL samples are decanted from the mercuric chloride preserved water analysis of nitrate,

phosphate and silicate by dispensing the sample via the 1/8” Teflon tube compression fitting by gentle pressure into 12mL HDPE tubes (Sarstedt 60.9922.241).

4 Quality control for the analysis of macronutrients

Macronutrient samples are analysed by CSIRO Hydrochemistry according to their standard operating procedures Hydrochem SOP 001-4 (Rees et al. 2018). In addition to supplying a log sheet with the samples, a best estimate of nutrient concentrations is provided so that calibration-standards concentrations can be adjusted accordingly, to better bracket the macronutrient concentration of the samples. CRM results are supplied with the sample results with a value and expanded uncertainty. The method measurement uncertainty is calculated for each nutrient based on the variation in the calibration curve, calibrations standards, pipette calibration and precision of the RMNS over time (Armishaw 2003).

Table 3. Estimated macronutrient uncertainties for RAS samples

Analyte	Calculated measurement uncertainty @1 uM	Method detection limits uM
Nitrate+nitrite (NOx)	±0.019	0.02
Silicate	±0.017	0.20
Phosphate	±0.024	0.02

Dissolved constituents are flagged 1 if the standards are in range, the CRM falls within control limits, duplicates are within the error for the method, and the dilution applied is regarded as good (flag 1). Poor duplicates are defined as falling outside of the range, defined as 1.96 times the standard deviation of the CRM. The mean value is flagged 2. If there is a failure at this point, there is the opportunity to resample from the archive.

The RAS results are compared with macronutrient bottle samples (in $\mu mol.kg^{-1}$) collected from casts at the SOTS site (an example for the most recent deployment is shown in Figure 1). Variations in the RAS sample data from the bottle data are revealed. Some of these are inevitable because of the general patchiness of the water caused by eddies, stratification, water mass intrusions etc, and the general mismatch in time. For this example, these variations were largest for the 'book-end' deployment and recovery voyages, while a rare voyage visiting the site during the deployment shows good agreement. Figure 2 compiles this information for the multi-year record. It also provides N/P and N/Si ratios to further evaluate sample integrity. More variable N/P values occur in the RAS samples and often as single sample spikes, possibly due to leaching of cell contents. The overall average N/P ratios are also lower in the RAS samples, and on this basis, we suspect that sample leaching compromises phosphate to some degree (by ~10%) in all RAS samples. Accordingly, all phosphate results are initially flagged 3 to indicate that they exhibit bias, although we consider that the seasonal structure reflects actual oceanographic conditions. Phosphate data greater than 2.5 umol kg^{-1}, the maximum value observed in bottle samples at the site, are downgraded to flag 4. In particular for Pulse-6 samples collected through filters. This filtration derived artefact also impacted silicate which are also flagged 4.

In the 2015-16 deployment of Pulse-11, unusually high N/Si ratios were observed. Examination of the individual N and Si seasonal records shows that they were similar to other years, but the Si numbers were particularly low in the last few samples when the concentrations were close to the detection limit (Table 2), and error propagation suggests that these high N/Si values result largely from this detection problem. Accordingly, no changes to N or Si flags were made for these samples.

The CTD comparisons also reveal that the small variation (<10 m) in actual depths of sampling by the RAS package on the Pulse moorings (as a result of the elastic tether responses to wave events – see the Annual Reports) were insufficient to have compromised any of the nutrient concentrations, i.e. the RAS remained within the well mixed surface layer at all times (Figures 3 and 4).

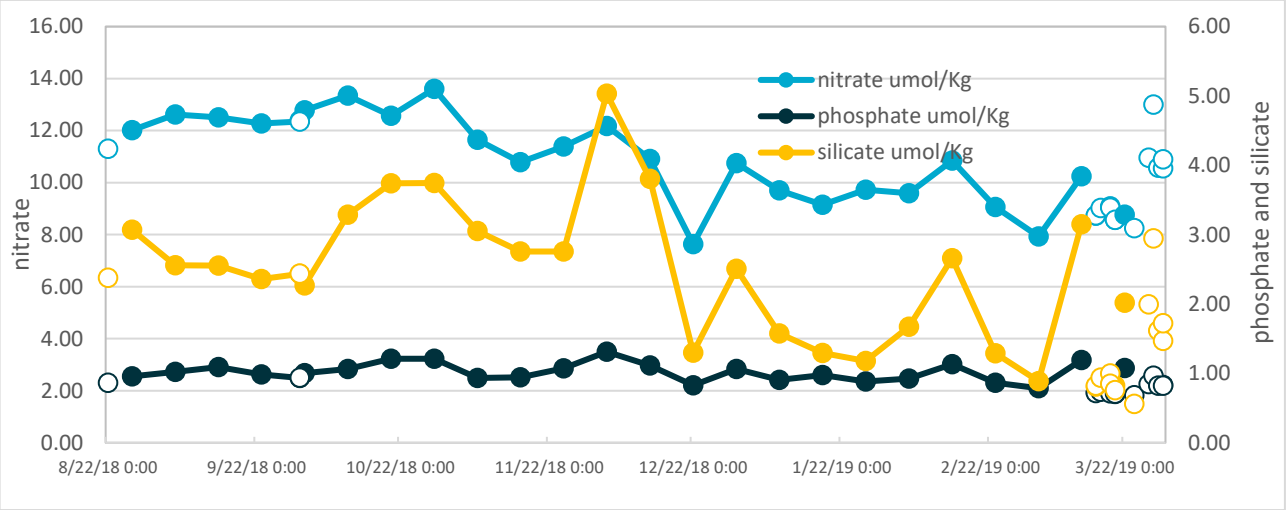

Figure 1. Samples recovered from RAS, SOFS7.5 2019 (solid points) showing macronutrients from Niskin bottles from casts adjacent the mooring (unfilled points) with an additional cast from voyage IN2018_V04

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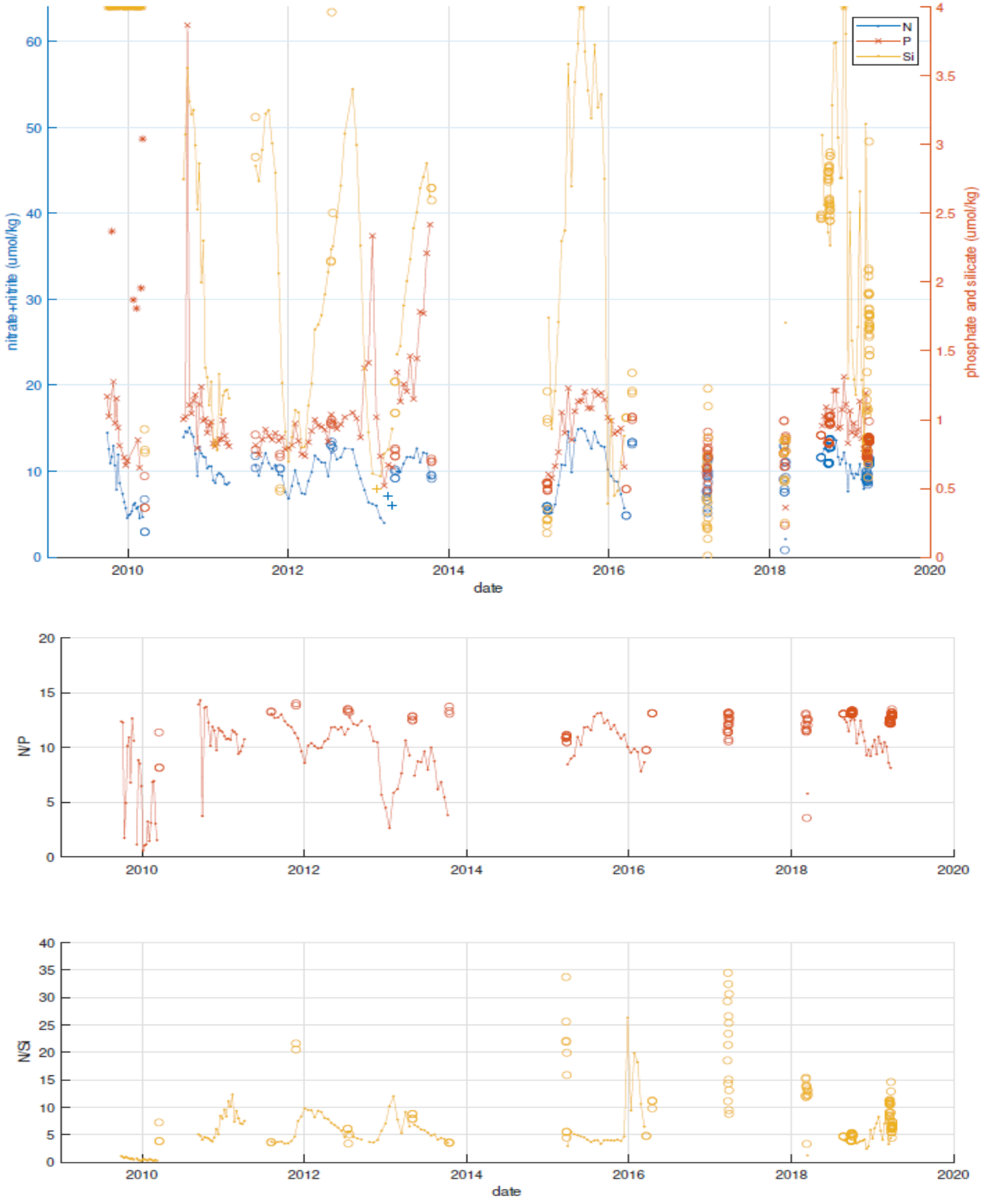

Figure 2. Multi-year comparison of RAS and Shipboard CTD bottle nutrient results: a. nitrate, phosphate and silicate concentrations, b. N/P molar ratios, c. N/Si molar ratios.

Note: that RAS concentrations are in µmol.kg^{-1} and CTD concentrations are in µmol L^{-1}(and thus the latter are ~3% higher because of this difference in units). Open circles are CTD data. RAS results are as follows: small points and lines are flagged 1 (good); + symbols flagged 2 (probably good), X are flagged 3 (probably bad), * are flagged 4 (bad).

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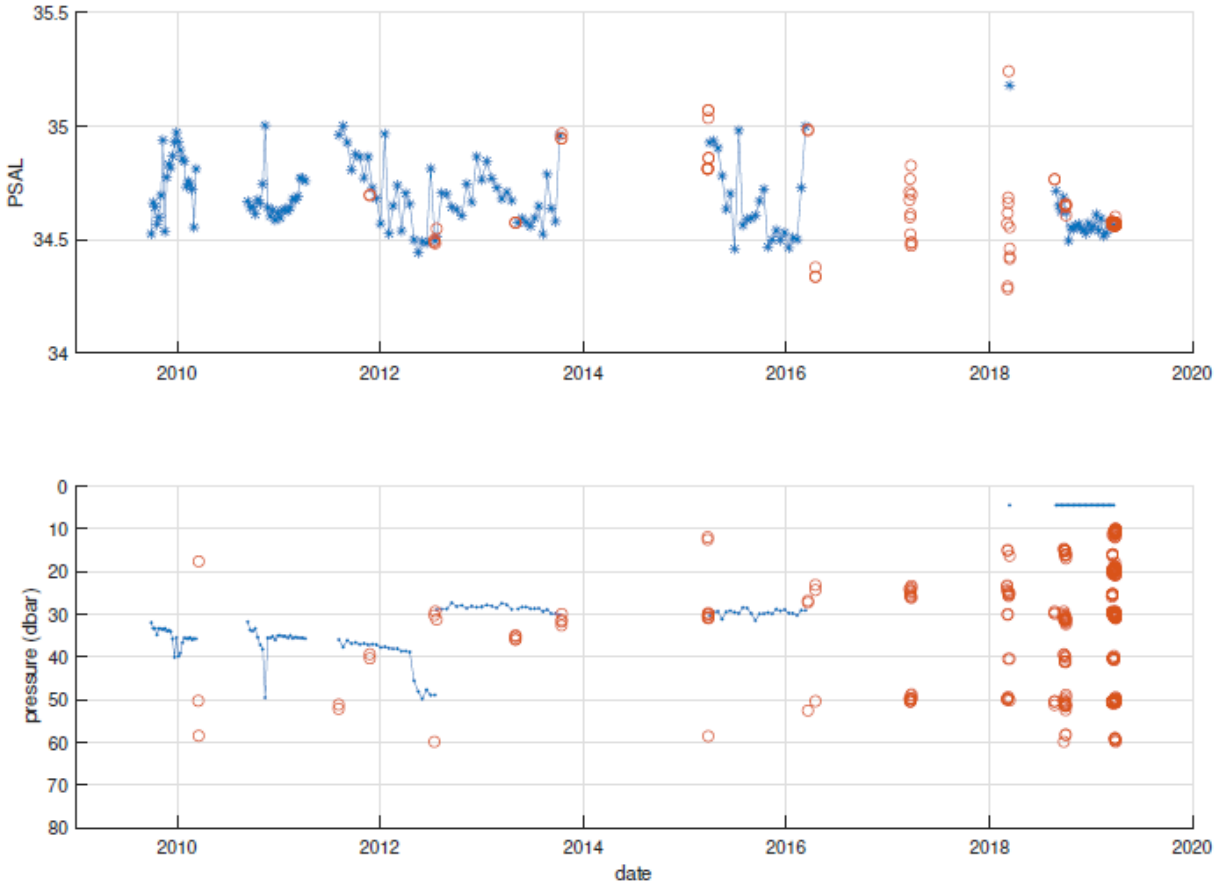

Figure 3. Multi-year comparison of RAS and Shipboard CTD bottles (open circles) a. salinities and b. depths of sampling

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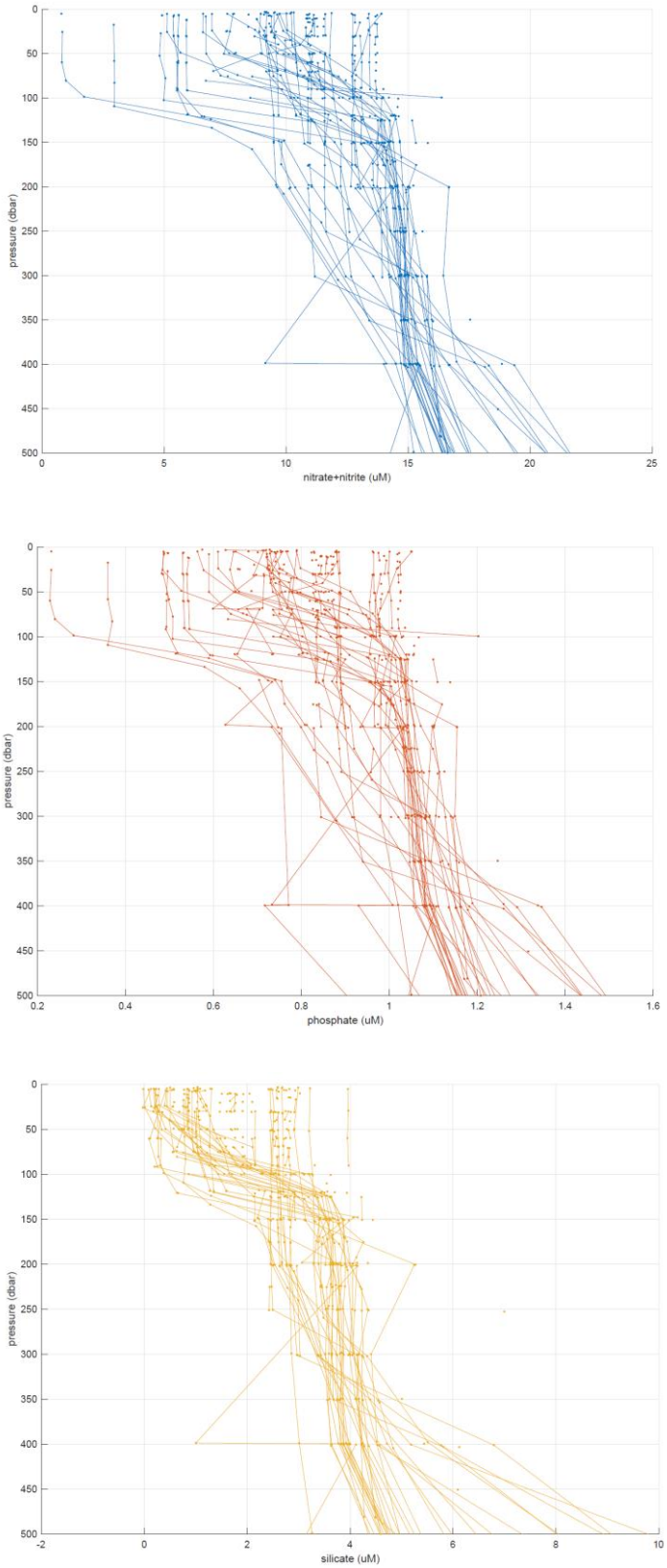

Figure 4. All upper ocean CTD bottle nutrient results versus depth for a. nitrate, b. phosphate and c. silicate. All concentrations are in µmol L^{-1}.

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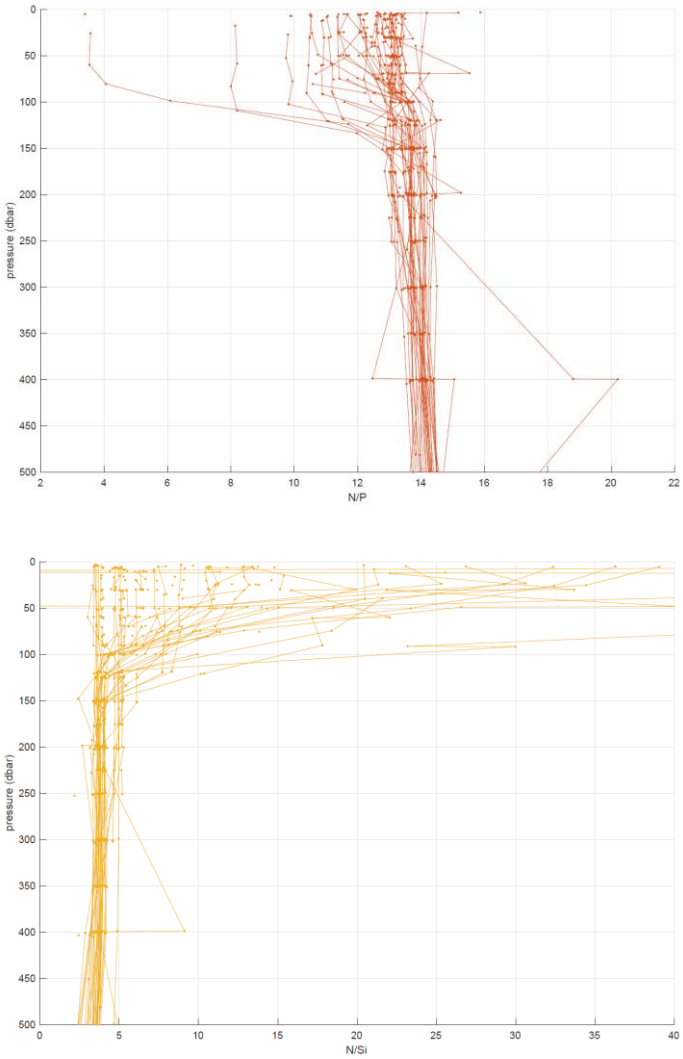

Figure 5. All upper ocean CTD bottle nutrient results versus depth for a. N/P molar ratio. b. N/Si molar ratio

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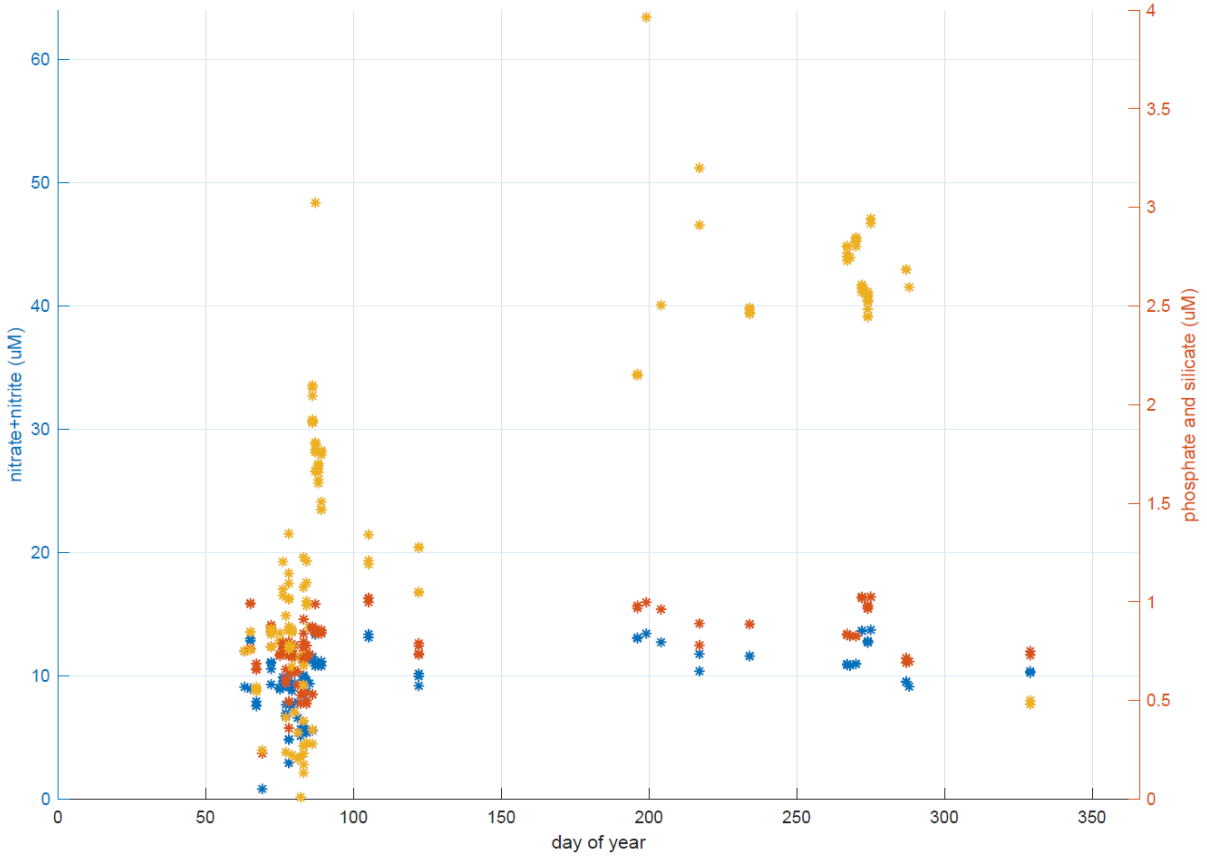

Figure 6. All upper ocean CTD bottle nutrient concentrations in depth range 10 – 60 dbar versus day of year (silicate=yellow). All concentrations are in µmol L^{-1}

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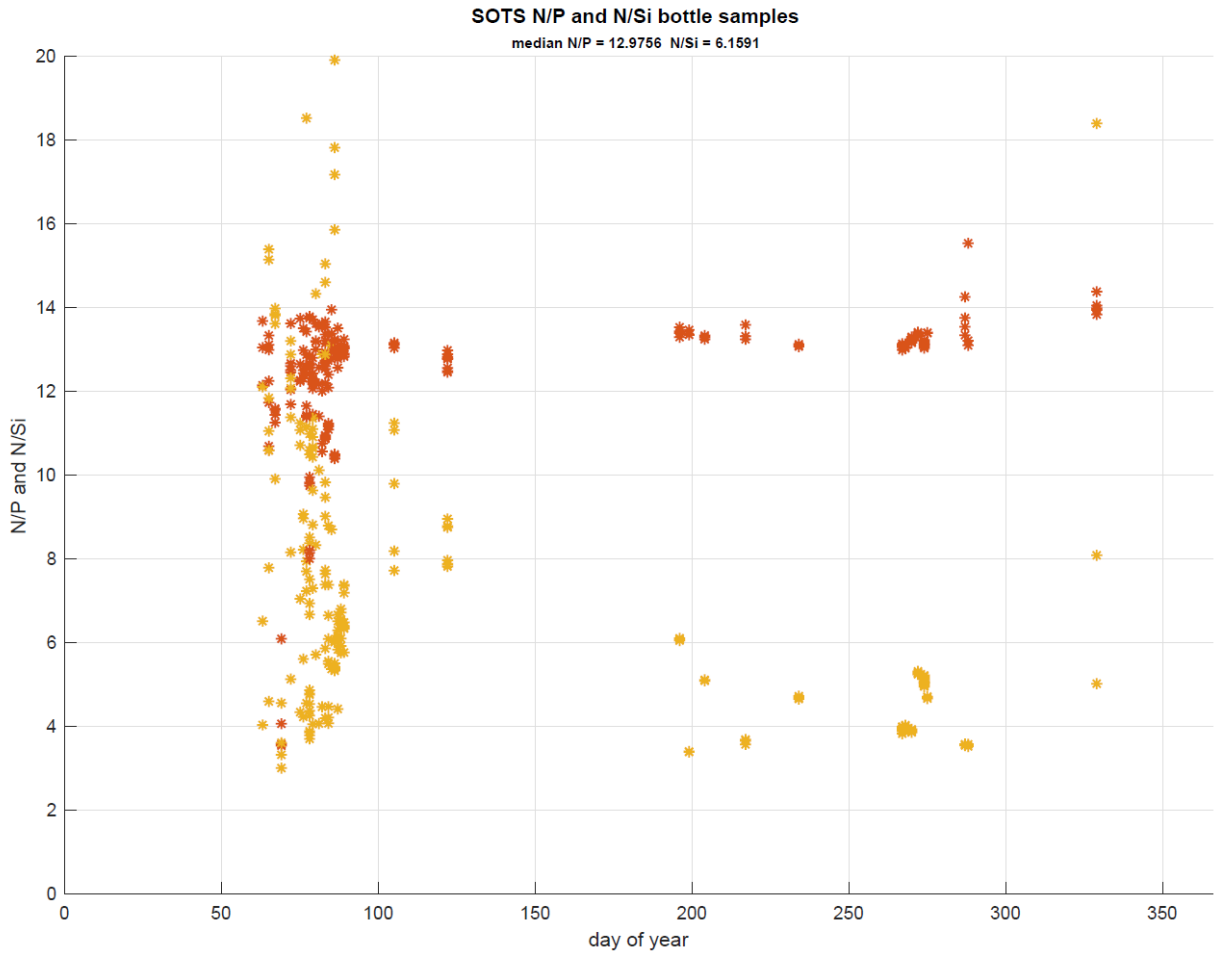

Figure 7. All upper ocean CTD bottle nutrient results versus depth for N/P (red) and N/Si (yellow) molar ratios in depth range 10 – 60 dbar versus day of year.

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5 Recommendations for Quality Assurance

Inclusion of low concentration calibration standards during the Hydrochemistry analyses should be continued. There is not yet any clear or simple way to reduce the phosphate release problem in the RAS samples. Implementing filtration with valve-isolated filtrate is a possible solution but it could potentially increase the pumping friction and reduce the delivered sample volume.

Reduction of sample volume requirements is a useful target for future efforts. Especially, because higher resolution sampling via single RAS samples (not paired) as recently programmed for SOFS-8 means less volume will be available at each time point. Discontinuation of the TCO_2 sampling (as a result of preliminary review that revealed persistently high TCO_2 problems) will provide sufficient volume to continue the macronutrient analyses as currently done. However, another method (e.g. Anton Parr density) will be required to determine sample salinity to derive the prime volume.

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