Calibration exercise for the Community Aquatic Monitoring Program (CAMP) nutrient analyses: establishing variability between filtered and unfiltered water samples and two analytical laboratories.

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Calibration exercise for the Community Aquatic Monitoring Program (CAMP) nutrient analyses: establishing variability between filtered and unfiltered water samples and two analytical laboratories.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDIX

ABSTRACT

Thériault, M.-H. and S.C. Courtenay. 2012. Calibration exercise for the Community Aquatic Monitoring Program (CAMP) nutrient analyses: establishing variability between filtered and unfiltered water samples and two analytical laboratories. Can. Tech. Rep. Fish. Aquat. Sci. 2980: ix + 29 p.

As part of the Community Aquatic Monitoring Program (CAMP) unfiltered water samples were collected between 2006 and 2008 and analyzed for dissolved inorganic nutrients (i.e., nitrate + nitrite ($NO_3 + NO_2$) subsequently referred to nitrate, nitrite (NO_2), ammonia (NH_3), orthophosphate (PO_4) subsequently referred to as phosphate, and silicate) by the Bedford Institute of Oceanography (BIO). In 2009 the nutrient component of CAMP was suspended because BIO could no longer process the samples. As a result, steps were undertaken to have the Maurice Lamontagne Institute (MLI) analyze future nutrient samples. MLI conducts analyses for nitrate, nitrite and phosphate, using similar equipment and methodologies as BIO, but recommends filtered samples whereas BIO recommends unfiltered samples. Therefore, before changing laboratories a calibration exercise between laboratories (BIO and MLI) and between filtered and unfiltered water samples was carried out in August 2009 with water samples collected from six CAMP sites. Results indicated that filtering produced significantly lower nutrient levels and analytical techniques at MLI produced significantly lower nutrient levels than BIO. These significant differences were caused by very few high nutrient values that skewed the data but were less pronounced when looking at the back-transformed data (from $\ln x + 0.1$). Furthermore, Pearson correlations between filtered and unfiltered samples and between laboratories were very high ($R = 0.79 - 0.99$) permitting the establishment of correction equations through linear regression. Linear regressions were also highly significant and adjusted R^2 ranged from 0.56 to 0.96. In conclusion, the two laboratories gave comparable results when low levels of nutrients were found in samples and conversion formulas were developed to facilitate relating the older data from BIO to the newer data from MLI.

RÉSUMÉ

Thériault, M.-H. & S.C. Courtenay. 2012. Exercice de calibration des éléments nutritifs pour le Programme Communautaire de Surveillance Aquatique (PCSA): évaluation de la variabilité entre des échantillons d'eau filtrés et non filtrés et entre deux laboratoires analytiques. Rapp. tech. can. sci. halieut. aquat. 2980: ix + 29 p.

Dans le cadre du Programme Communautaire de Surveillance Aquatique (PCSA), des échantillons d'eau non filtrés ont été recueillis entre 2006 et 2008 et analysés par l'Institue d'Océanographie de Bedford (IOB) pour quantifier les niveaux d'éléments nutritifs inorganiques dissous dans l'eau (i.e., nitrate+nitrite (NO3 + NO2) subséquemment référé à nitrate dans ce rapport, nitrite (NO2), ammoniaque (NH3), orthophosphate subséquemment référé à phosphate et silicate). En 2009, les analyses d'éléments nutritifs du PCSA furent suspendues puisque l'IOB ne pouvait plus analyser nos échantillons. L'institut Maurice-Lamontagne (IML) fut donc approché pour entreprendre ces analyses dans le futur. L'institue Maurice-Lamontagne effectue des analyses pour le nitrate, nitrite et phosphate en utilisant un équipement et une méthodologie similaire à IOB mais recommande de filtrer les échantillons tandis que IOB recommande de ne pas filtrer les échantillons. Avant de changer de laboratoire, un exercice de calibration pour évaluer si des différences existaient entre les deux laboratoires (IOB et IML) et entre les échantillons d'eau filtrés et non filtrés fut entreprit à six sites du PCSA en août 2009. Les résultats indiquent que la filtration semble avoir diminué significativement le niveau d'éléments nutritifs et que l'IML ait trouvé des résultats significativement inférieurs comparés à l'IOB. Ces différences significatives furent générale causé par quelques données très élevées biaisant ainsi la base de données. En regardant plutôt aux données transformées (ln x + 0.1) ces différences sont moins prononcées. Par ailleurs, les corrélations entre les échantillons filtrés et non filtrés et entre les échantillons analysés aux deux laboratoires étaient très élevés (R varient entre 0.79 à 0.99) nous permettant ainsi d'établir des équations de corrections à l'aide de régressions linéaires. Les régressions linéaires étaient tous significatif et les R2 ajusté tous assez élevés (R2 ajusté varient entre 0.56 to 0.96). En conclusion, les deux laboratoires ont donnés des résultats comparables dans les échantillons à faible niveaux de nutriments et des formules de conversion ont été développées pour faciliter la comparaison entre les données recueillis depuis 2006 analysées par IOB et les nouvelles données analysées par IML.

INTRODUCTION

As the global population climbs and coastlines are inundated with human activities, the problem of nutrient enrichment in coastal waters grows. Nutrient enrichment can cause numerous changes to the ecosystem such as: increased plant growth, development of harmful algal blooms, decrease in levels of dissolved oxygen (DO), increased organic matter in the sediment, hypoxic or anoxic conditions due to the bacterial decomposition of macrophytes causing further depletion of DO that may in turn result in the death of benthic organisms and in severe cases, fish kills (CCME 2007). Nutrient enrichment has been reported in many watersheds in the southern Gulf of St. Lawrence (sGSL) and has been linked, for example, to high levels of agricultural activities in Prince Edward Island (PEI) (Raymond and al. 2002) and to fish processing plants in New Brunswick (NB) (Roy Consultants Ltd. et al. 2003). Because the Community Aquatic Monitoring Program (CAMP) was already established in many estuaries and bays of the sGSL, it was suggested that a nutrient component be added to the program. Nutrient sampling was incorporated in the CAMP protocol for the first time in 2006. These data benefit CAMP by adding additional information with which to understand the community structure and establish the health status of our coastal ecosystems.

Two water samples per station were collected in September 2006 and a complete monthly sampling from May to September was carried out in 2007 and 2008. The water samples were analyzed by the nutrient laboratory at the Bedford Institute of Oceanography (BIO) for silicate, orthophosphate (subsequently referred to phosphate in this report), nitrate + nitrite (subsequently referred to nitrate in this report), nitrite and ammonia using colorimetric techniques on a Technicon Autoanalyzer II segmented flow analyzer (details of the methodology are given in Strain and Clement, 1996).

In 2009, the nutrient component of CAMP was suspended because BIO could no longer process the samples. As a result, steps were undertaken to have the Maurice Lamontagne Institute (MLI) analyze the nutrient samples. However, before having a new laboratory

analyze our samples, it was suggested that a calibration exercise be undertaken between the two laboratories (BIO and MLI).

A second issue with changing analytical laboratories concerned filtration of the water samples before analyses. The methodology used by BIO from 2006 to 2008 did not require filtration of the water samples prior to the dissolved inorganic nutrient analyses unless water samples were very muddy. The nutrient sampling protocol of the Atlantic Zonal Monitoring Program also does not call for filtering, unless samples have significant sediment load, to minimize sample contamination (Mitchell et al. 2002). The thinking at BIO was that filtering was likely to introduce more problems than it solved for oceanic samples but this may not be the case for estuarine samples which can have much higher nutrient values and particulate matter. In contrast, the methodology employed at the MLI laboratory suggested that samples be filtered in the field before being analyzed. Filtration increases preservation qualities and removes large particles (sediment, organic matter, phytoplankton and zooplankton) which can decrease the precision and the accuracy of the analyses for dissolved inorganic nutrients. Unfiltered water samples are usually used to determine total nitrogen (TN) and total phosphorus (TP), treated first by chemical digestion. Because BIO and MLI did not include a chemical digestion step in their process they do not determine TN and TP, but rather total dissolved inorganic nutrient which is usually analyzed from filtered water. For example, filtered water samples were used to measure inorganic nutrients to determine indices of coastal-zone eutrophication (Ryan et al. 2008) and to measure dissolved inorganic nitrogen in sea water (Sharp et al. 2002). On the other hand, Strain and Yeats (1999) used unfiltered water samples to determine the relationship between dissolved inorganic nutrients and eutrophication in inlets located in eastern Canada and to measure dissolved nutrients in Prince Edward Island inlets (Bates and Yeats 2006). Therefore, dissolved inorganic nutrient analyses for coastal waters do not have standardized protocols concerning filtration. To facilitate comparisons of CAMP water nutrient data from MLI which filters water, and BIO which does not, we examined the influence of filtering. This report describes the results of this calibration exercise.

METHODS AND MATERIALS

Site selection

From August 17-19 2009, filtered and unfiltered water samples were collected at six CAMP sites. Two CAMP sites per province were chosen to represent all three provinces (NB, PEI and Nova Scotia (NS)) participating in CAMP. Other criteria in site selection included proximity to the Gulf Fisheries Centre (GFC) and easy accessibility to the station. The six sites selected were: Bouctouche Harbour and Scoudouc River in NB, Trout River and Summerside in PEI, Pugwash estuary and Pictou Harbour in NS (Fig. 1). Water samples in NB were sampled on August 17th 2009 between 10:50h and 16:30h around low tide. Water samples in PEI were collected on August 18th 2009 between 10:55h and 17:05h around low tide. Water samples in NS were collected on August 19th 2009 between 10:30h and 18:15h and around low tide in Pugwash but around high tide in Pictou. Due to logistics we were not able to sample Pictou at low tide as we did for the other sites. All samples were collected by Marie-Hélène Thériault for consistency.

Methodology

Before going to the field, all 60 ml Luer-Lok syringes (60 CC BD, non-sterile Luer Lok syringes from Cole Parmer cat # RK-07945-28) used for filtering and 15 ml polypropylene centrifuge tubes with flat cap (VWR cat # 89004-368) used for collecting the water samples for MLI were dipped in a 5% v/v hydrochloric acid bath for 2 days and rinsed three times with deionized water. Similar treatments were done at BIO for the 30 ml wide-mouth Nalgene bottles used to collect the BIO samples. Duplicate water bottles were identified with a six digit number.

In the field eight water samples per station were collected (duplicate filtered and unfiltered for each of the two laboratories; 2 duplicates x 2 methodologies (filtered and unfiltered) x 2 laboratories (BIO and MLI) = 8 bottles). All water samples were collected wearing polyethylene gloves to avoid contamination. With a pair of waders, the biologist walked into the estuary from shore to 60-100 cm depth. Then two unfiltered water samples for BIO and two for MLI were collected from mid-water depth (~30 to 50 cm from the water surface). Each bottle was rinsed with estuarine water three times and then filled leaving a space to allow water to expand when freezing. Before returning to shore,

a 1L acid washed Nalgene bottle was rinsed three times and then filled with estuarine water from the same area sampled for un-filtered water. This water was then used to collect filtered water samples. To filter the water samples on the field, a Luer-Lok syringe and a 25 mm diameter polyethersulfone filter with 0.45µm pore size (VWR cat # CA28145-503) was used. The syringe was first rinsed twice with the water and then filled with 60 ml of the test water. The filter was then installed on the tip of the syringe and rinsed by squeezing approximately 10 ml of water through it. Two 15ml centrifuge tubes were rinsed once and then 80% filled with filtered water for the MLI analyses. A new filter was installed on the same syringe to collect filtered water for BIO. The filter and two 30 ml Nalgene bottles were rinsed once with filtered water and then filled with 15 - 20 ml filtered water for BIO. All bottles were then closed tightly, placed directly on crushed ice and frozen at -20ºC once back at GFC. At each station a new syringe was used and a new filter was also used between laboratories to reduce contamination. The 1L Nalgene bottle used to collect water for filtered samples was also rinsed twice between stations with deionized water.

In September, water samples on dry ice were driven to MLI and placed in a freezer at -80ºC immediately upon arrival. Water samples on ice packs were driven to BIO and placed in a freezer at -20ºC upon arrival.

Laboratory analysis

Bedford Institute of Oceanography (BIO)

At BIO, the water samples were analyzed for silicate, orthophosphate (PO_4) (subsequently referred to phosphate in this report), nitrate + nitrite ($NO_2 + NO_3$) (subsequently referred to nitrate in this report), nitrite (NO_2) and ammonia (NH_3) using colorimetric techniques on a Technicon AutoAnalyzer II segmented flow analyzer (details of the methods are given in Strain and Clement, 1996, available on line at: http://www.dfo-mpo.gc.ca/Library/208778.pdf). All nutrient data in this report are in units of micromoles per liter of water (µM). Calibrations were done using a series of standards at six different concentrations analyzed at the beginning and end of each

AutoAnalyzer run (Strain and Clement, 1996). All standards and samples were analyzed in duplicate. A natural sea water sample (MOOS-1) produced by the National Research Council (NRC) of Canada was then used to verify the calibration. Note that the AutoAnalyzer cannot process water samples that are too colored. Therefore, BIO was not able to process the unfiltered duplicate samples from Pictou station 5 (Boat Harbour station located near the discharge of treated pulp mill effluent) because the samples were too colored (brown). It's also important to note that the protocol used to analyze the water samples for this experiment was for low concentration nutrient samples such as those collected at sea. A different protocol for samples collected at discharge sites could have been used but was not for this experiment. All water samples at both laboratories were analyzed using the same chemical process for low concentration seawater samples.

Maurice Lamontagne Institute (MLI)

At MLI, the water samples were analyzed only for phosphate, nitrate + nitrite (subsequently referred to nitrate in this report) and nitrite using the same methodology as BIO (Strain and Clement, 1996). The samples were processed using a more recent AutoAnalyzer III from Bran & Luebbe. At MLI, calibrations were done using a series of standards at 6 different concentrations. MLI also used the MOOS standard to verify their calibration and other standards such as Ricca Chemical Co standards from Arlington Texas and CSK standards from the Sagami Research Center in Japan. Maurice Lamontagne Institute also prepared in-house standard samples at two different concentrations with natural seawater. All these samples were used on a daily basis to verify calibrations.

Statistical Analyses

Results from duplicate water samples collected at each station were averaged to provide a single, best measure of each nutrient to carry forward in the analyses (Appendix 1). Data were verified for normality with probability plots prior to analysis. All data were natural log transformed ($\ln x + 0.1$) to improve normality. Only nitrate ($NO_3 + NO_2$), phosphate

(PO_4) and nitrite (NO_2) were used in the inter-laboratory comparison since these are the three nutrients analyzed by both laboratories. Because we wanted to test the difference between both treatments (i.e. treatment 1 = filtered and unfiltered water samples; treatment 2 = BIO and MLI), and not sites or stations, a randomized block analysis of variance design was used. Thirty-three to thirty-four stations (6 sites x 6 stations – 2 stations not sampled in Pugwash estuary due to an extremely low tide and 1 unfiltered sample in Pictou station 5 not analyzed by BIO because sample was too colored) were used as blocks to compare each individual nutrient value between filtered and unfiltered and between laboratories. Pearson correlation coefficients for comparing nutrient levels between laboratories and filtration process were also calculated. Equations relating MLI data to BIO data were calculated by linear regression. Univariate statistical analyses were performed using the Systat version 11 software. The level of significance was set at $P < 0.05$.

RESULTS AND DISCUSSION

Significant differences were detected between filtered and unfiltered samples and between samples analyzed at the two laboratories for concentrations of nitrite and phosphate (see Table 1 for randomized block design ANOVA results). Marginally significant differences were found for nitrate (Filtration: $F_{1,98} = 3.83$, $p = 0.05$ and Laboratory: $F_{1,98} = 4.45$, $p = 0.04$).

To evaluate how large these significant differences were, we calculated the percent differences ({1-(smallest value/largest value)} x 100) between laboratories and between filtering processes (Appendix 2). We decided to express the differences between laboratories and filtered/unfiltered samples as percentage rather than absolute differences because a 1 µM error is quite different for a 2 µM sample than a 200 µM sample and because we have samples that range greatly in nutrient values. Percent differences for nitrate levels in filtered samples analyzed by MLI and BIO ranged from 2 to 80% and largest percent differences were usually found in samples with very high nitrate values (e.g., Summerside station 6; Appendix 2). Table 2 shows that on average percent difference between filtered/unfiltered samples were smaller (26%) than between

laboratories (33%). When comparing differences between laboratories, differences were on average larger between filtered samples (37%) than unfiltered samples (29%) (Table 2). The filtration process gave higher percent difference which could be explained by possible contamination associated with the greater manipulation of the sample. When comparing differences between filtered/unfiltered samples for each laboratory individually, samples processed at BIO gave on average 21% difference and MLI 32% difference. On average differences between laboratory and filtering process were quite large (15 to 43%) depending on the nutrient, but largest disagreements between laboratories and filtering processes were usually observed with highest nutrient levels and smaller percent difference was generally observed at low nutrient levels. Furthermore, the percent differences in Appendix 2 are skewed by a few very high values. When extreme data are down-weighted by natural log (x+0.1) transformation, the probability curves are greatly improved and we get a more representative mean. When we calculate the back-transformed (from ln x+0.1) nitrate, nitrite and phosphate levels (μM) in water samples it reduces the effect of these very large values. Therefore, for the majority of samples both laboratories and both filtering process gave comparable results as is reflected in the similar back-transformed means and confidence intervals in Tables 3 to 5. Filtered samples generally (86% of the time considering all nutrients and both labs) showed lower nutrient levels than unfiltered samples, although the reverse was observed in a few cases possibly due to contamination, and measurements made at MLI were generally slightly lower than those measured at BIO (Table 3 to 5).

Pearson correlations were very high between filtered and unfiltered samples and between samples processed at the two laboratories (R = 0.79 to 0.99 depending on nutrient and filtering; Table 6). This means that corrections can be calculated and applied to the nutrient data we have collected in the CAMP program since September 2006 (i.e., unfiltered and processed at BIO) for direct comparison to data that will be produced by MLI (i.e., filtered samples). Table 7 show that linear regressions for nitrite was highest with an adjusted R^2 value equal to 0.96 followed by nitrate ($R^2 = 0.79$) and phosphate had the smallest adjusted $R^2 = 0.56$. All 3 regressions were highly significant ($P < 0.001$). Multiple linear regressions could have been calculated but we concentrated on the

regression equation that would be needed to convert old data analyzed at BIO (i.e., unfiltered samples) to new IML filtered data. Table 7 gives the correction formulas for all three nutrients and facilitates the retention of the BIO nutrient data collected from 2006 to 2008 within the CAMP nutrient database to be continued through IML analyses.

High nutrient levels were found at four stations which were located near anthropogenic sources: station 6 in Summerside (PEI), station 2 in Trout River (PEI), station 4 in Scoudouc (NB) and station 4 in Bouctouche (NB). Station 6 in Summerside is located near a large potato processing facility and cottages with septic fields which could account for the high nitrate loads (BIO filtered sample=35 µM; BIO unfiltered sample=35 µM; MLI filtered sample=178 µM; MLI unfiltered sample=162 µM; Appendix 2). In addition, there are 45 degree banks behind this station which would contribute to rapid runoff in that area. Station 2 in Trout River is the most upstream station and is known to have high nitrogen loading and excessive sea lettuce growth, possibly resulting from agricultural runoff located in the watershed and/or because of low flushing rate in this part of the river (Schein et al. 2011). Station 4 in Scoudouc River is located next to a lobster processing plant which discharges its waste water through an effluent pipe into the sampling area (Thériault et al. 2007). Effluent was being discharged the day water samples were collected which could explain the high phosphate levels found by BIO (filtered: 85 µM and unfiltered: 31 µM) and MLI (filtered: 32 µM and unfiltered: 31 µM; Appendix 2). Effluents of fish processing plants can contain high phosphorus concentrations from the blood and carapaces of the crustaceans (NovaTec Consultants Inc. 1994). Finally, station 4 in Bouctouche is located approximately 500m downstream of the sewage treatment plant which might explain the high phosphate level found there by BIO (filtered sample: 37 µM and unfiltered sample: 63 µM) and MLI (filtered sample:2 µM and unfiltered sample: 4 µM).

CONCLUSIONS

Our analyses indicate that while filtering may produce significantly lower nutrient levels and analytical techniques at MLI may produce significantly lower nutrient levels than BIO, correlation and linear regressions were high and significant. Largest disagreements between laboratories and filtering processes were observed with highest nutrient levels and smaller differences were generally observed at low nutrient levels. Even though significant differences were found, when we take out the high nutrient values which show a lot of variance, the differences become small. The samples with high nutrient values located near anthropogenic discharges could have been analyzed through a different procedure but were not. For this experiment all samples used the Atlantic Zonal Monitoring Program Sampling Protocol which is usually applied to seawater samples (i.e., samples of low nutrient concentrations). Other sources of variation could have been introduced including use of different standard solutions for calibration and chemical reagents. In addition, for the analysis of nitrate, the condition of the cadmium column in the auto-analyzer could also contribute to small variations in nitrate readings (M-L Dubé, Institut Maurice-Lamontagne, Mont-Joli Québec, G5H 3Z4, pers. comm.). Furthermore, all samples for subsequent sub-sampling should have been collected from a single large homogeneous sample instead of collecting several water samples from the same "station" as was done in the present study. For this exercise, the unfiltered samples were drawn directly from the estuary and the filtered samples were drawn from a homogeneous sample taken from the estuary. This methodology possibly introduced another source of variance between the samples as there can be strong horizontal gradients near point sources. Collectively, these small variations could explain why MLI found slightly lower nutrient levels than BIO. Therefore, conversion factors were derived through linear regression to facilitate comparison of 2006 to 2008 data generated by BIO to the newer IML data (see Table 7 for regression equations). The data obtained from 2006 to 2008 with unfiltered samples processed at BIO will be comparable with the data provided in 2010 and 2011 by MLI by applying this conversion factor. Thus, the nutrient data collected by CAMP to date are valuable and should be properly archived.

This small scale calibration exercise also demonstrated that filtration of water samples only slightly altered the quantification of nutrients. Filtering water samples resulted in an average 26% difference in nutrient concentrations. Although there doesn't seem to be a common practice among studies to determine dissolved inorganic nutrients in estuaries, most recent studies carried out in the Atlantic region have not filtered their water samples (Stain and Yeats 1999; Strain et al. 2001; Bates and Yeats, 2002), while most studies in the literature have (Brown & Ozretich, 2009; Durisch-Kaiser et al. 2010; Fulweiler et al. 2010; Luo et al. 2010). Even though the two laboratories differed in their advice on the need for field-filtering a recent inter-laboratory calibration exercise for dissolved inorganic nutrients in seawater coordinated by the National Research Council of Canada showed MLI (lab #23) and BIO (lab #10) to be among the best-performing laboratories, giving results comparable to the assigned mean (Willie and Clancy 2002). This further demonstrates that both laboratories produce accurate and comparable data.

In conclusion, both laboratories use traditional colorimetric procedures and equipment to analyze dissolved inorganic nutrient in water and produced comparable results when low levels of nutrients were found in samples. The protocol that MLI follows is to filter water samples so we recommend that CAMP follow this protocol for the present, while acknowledging that it will not make much difference except for samples of very high nutrient values. A future experiment could compare protocols used to analyze high nutrient concentration samples from discharge areas versus the protocol used for low nutrient concentration found in sea water to determine whether modified protocols are merited for highly impacted samples.

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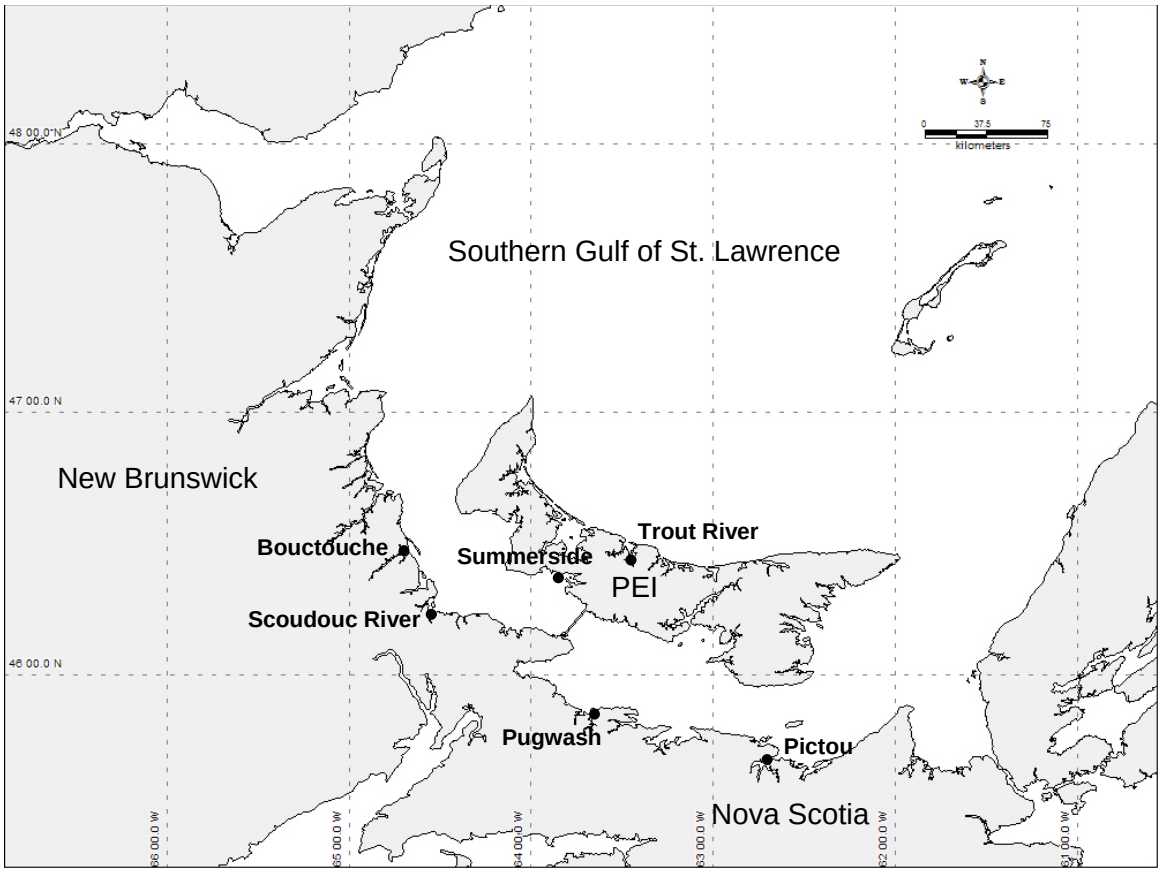

Figure 1: Location of the six CAMP sites where water samples were collected in August 2009 for the calibration exercise.

Table 1: Randomized block design analysis of variance results for nitrate ($NO_3 + NO_2$), nitrite (NO_2) and phosphate (PO_4). Treatment 1 indicates differences between filtered and unfiltered data. Treatment 2 indicates differences between MLI and BIO.

Analysis of Variance for nitrate

Source	Sum-of-Squares	df	Mean-Square	F-ratio	P
Block	320.557	33	9.714	102.563	0.000
Treat 1=Filtered	0.363	1	0.363	3.831	0.053
Treat 2=Lab	0.421	1	0.421	4.449	**0.037**
Treat 1 x Treat 2	0.059	1	0.059	0.618	0.434
Error	9.282	98	0.095		

Analysis of Variance for nitrite

Source	Sum-of-Squares	df	Mean-Square	F-ratio	P
Block	84.265	33	2.553	56.758	0.000
Treat 1=Filtered	1.400	1	1.400	31.108	**0.000**
Treat 2=Lab	0.796	1	0.796	17.683	**0.000**
Treat 1 x Treat 2	0.306	1	0.306	6.797	**0.011**
Error	4.409	98	0.045		

Analysis of Variance for phosphate

Source	Sum-of-Squares	df	Mean-Square	F-ratio	P
Block	107.686	33	3.263	21.485	0.000
Treat1=Filtered	4.047	1	4.047	26.643	**0.000**
Treat 2=Lab	3.201	1	3.201	21.072	**0.000**
Treat 1 x Treat 2	0.024	1	0.024	0.159	0.691
Error	14.885	98	0.152		

Table 2: Average percent difference between BIO and MLI and between filtered (F) and unfiltered (UF) samples for each of the three nutrients analyzed. The percent difference was calculated as follow: {1-(smallest value/largest value)} x 100.

	Average % difference			
	Laboratory (BIO vs. MLI)		**Filtration (F vs. UF)**	
Nutrient	Filtered	Unfiltered	BIO	MLI
Nitrate	33.31%	34.41%	14.70%	19.21%
Nitrite	43.25%	27.78%	17.83%	39.94%
Phosphate	33.31%	24.79%	31.03%	35.49%
Average	**36.62%**	**28.99%**	**21.19%**	**31.55%**
Grand Average	**32.81%**		**26.37%**	

Table 3: Back-transformed (from ln (x+0.1)) nitrate levels (µM) in water samples collected from 33-34 stations distributed among 6 CAMP sites (4-6 stations per site).

Lab	Nitrate	Filtered	Unfiltered
BIO	Mean	1.035	1.131
	95%CI	0.598-1.748	0.639-1.954
	N	34	33
MLI	Mean	0.873	1.026
	95%CI	0.433-1.677	0.512-1.971
	N	34	34

Table 4: Back-transformed (from ln (x+0.1)) nitrite levels (µM) in water samples collected from 33-34 stations distributed among 6 CAMP sites (4-6 stations per site).

Lab	Nitrite	Filtered	Unfiltered
BIO	Mean	0.244	0.276
	95%CI	0.169-0.340	0.195-0.379
	N	34	33
MLI	Mean	0.168	0.262
	95%CI	0.095-0.269	0.160-0.404
	N	34	34

Table 5: Back-transformed (from ln(x+0.1)) phosphate levels (µM) in water samples collected from 33-34 stations distributed among 6 CAMP sites (4-6 stations per site).

Lab	Phosphate	Filtered	Unfiltered
BIO	Mean	0.968	1.370
	95%CI	0.626-1.473	0.924-2.011
	N	34	33
MLI	Mean	0.664	1.011
	95%CI	0.457-0.949	0.738-1.371
	N	34	34

Table 6: Pearson correlation coefficients for nutrient levels in water samples either filtered upon collection or not filtered, then frozen and subsequently analyzed at BIO or IML. Duplicate samples were collected from 6 stations within each of 6 CAMP sites (2 from each of NB, NS and PEI). Nutrient levels from the two duplicate samples collected at each station were averaged for analysis. Two stations could not be sampled at one NS site (Pugwash) due to extreme low tide and BIO did not process one unfiltered sample from Pictou Station 5 (site of pulp mill discharge). With these omissions, sample size for comparisons was 33 or 34. Data were all ln (X+0.1) transformed to improve normality. **All correlations are highly significant (P<0.001).**

Nutrient	Comparison	R	N
Nitrate	Filtered BIO vs IML	0.896	34
	Unfiltered BIO vs IML	0.891	33
	BIO Filtered vs Unfiltered	0.988	33
	IML Filtered vs Unfiltered	0.994	34
Nitrite	Filtered BIO vs IML	0.869	34
	Unfiltered BIO vs IML	0.968	33
	BIO Filtered vs Unfiltered	0.989	33
	IML Filtered vs Unfiltered	0.949	34
Phosphate	Filtered BIO vs IML	0.897	34
	Unfiltered BIO vs IML	0.791	33
	BIO Filtered vs Unfiltered	0.891	33
	IML Filtered vs Unfiltered	0.899	34

Table 7: Linear regressions for nutrient levels in water samples either filtered upon collection or not filtered, then frozen and subsequently analyzed at BIO or MLI. Duplicate samples were collected from 6 stations within each of 6 CAMP sites. Nutrient levels from the two duplicate samples collected at each station were averaged for analysis. Two stations could not be sampled at one NS site (Pugwash) due to extreme low tide and BIO did not process one unfiltered sample from Pictou Station 5 (site of pulp mill discharge). With these omissions, sample size for comparisons was 33. Data were all ln (X+0.1) transformed to improve normality. **All linear regressions are highly significant (P<0.001).**

Nutrient	Linear Regression Equation	Adjusted R^2	N	$F_{(1,33)}$
Nitrate	Ln(Nitrate + 0.1) IML filtered = 1.080 Ln (Nitrate + 0.1) BIO unfiltered - 0.226	0.788	33	120.11
Nitrite	Ln(Nitrite + 0.1) IML filtered = 1.334Ln(Nitrite + 0.1) BIO unfiltered – 0.020	0.961	33	792.98
Phosphate	Ln(Phosphate+0.1) IML filtered = 0.684 Ln (Phosphate+0.1) BIO unfiltered – 0.527	0.557	33	41.27

Appendix 1: Water sample results from the 2009 calibration exercise for CAMP nutrient sampling. Data from each duplicate sample collected at each station and absolute differences between each duplicate value are included in this appendix. All data are in µM.

Laboratory	Rep1 Nitrate	Rep2 Nitrate	Nitrate absolute difference	Rep1 Phosphate	Rep2 Phosphate	Phosphate absolute difference	Rep1 Nitrite	Rep2 Nitrite	Nitrite absolute difference
BIO	0.90	1.09	**0.19**	0.67	0.60	**0.06**	0.20	0.23	**0.04**
BIO	2.21	2.32	**0.11**	1.35	0.58	**0.77**	0.26	0.22	**0.04**
BIO	0.29	0.28	**0.02**	0.90	0.90	**0.00**	0.15	0.15	**0.00**
BIO	1.99	2.08	**0.09**	31.80	29.80	**2.00**	0.65	0.73	**0.08**
BIO	1.14	1.36	**0.21**	1.42	1.45	**0.03**	0.36	0.37	**0.01**
BIO	0.68	0.65	**0.03**	0.91	0.85	**0.06**	0.24	0.24	**0.01**
BIO	0.52	0.52	**0.01**	0.64	0.61	**0.03**	0.17	0.16	**0.01**
BIO	2.39	2.41	**0.02**	0.44	0.45	**0.00**	0.19	0.20	**0.01**
BIO	0.33	0.33	**0.00**	0.54	0.53	**0.01**	0.16	0.16	**0.00**
BIO	1.63	1.52	**0.10**	99.00	71.20	**27.80**	0.36	0.36	**0.01**
BIO	1.20	1.22	**0.02**	1.21	1.27	**0.06**	0.31	0.31	**0.01**
BIO	0.53	0.55	**0.01**	0.70	0.60	**0.10**	0.19	0.20	**0.00**
BIO	0.28	0.27	**0.02**	1.39	1.59	**0.20**	0.15	0.13	**0.02**
BIO	0.26	0.28	**0.02**	1.76	1.78	**0.02**	0.12	0.12	**0.00**
BIO	0.80	0.80	**0.00**	1.89	1.89	**0.00**	0.16	0.17	**0.01**
BIO	20.66	16.46	**4.20**	75.00	50.20	**24.80**	2.62	2.62	**0.00**
BIO	0.44	0.39	**0.05**	1.27	1.38	**0.12**	0.23	0.19	**0.05**
BIO	0.24	0.21	**0.03**	1.29	1.47	**0.18**	0.11	0.10	**0.01**
BIO	0.24	0.24	**0.01**	1.20	1.11	**0.09**	0.12	0.11	**0.01**
BIO	0.26	0.25	**0.01**	1.54	1.58	**0.04**	0.11	0.11	**0.00**
BIO	0.94	0.94	**0.01**	1.74	1.74	**0.00**	0.16	0.16	**0.00**
BIO	10.32	10.25	**0.07**	36.80	36.40	**0.40**	2.61	2.61	**0.00**
BIO	0.40	0.36	**0.04**	1.30	1.24	**0.07**	0.19	0.15	**0.03**

Laboratory	Rep1 Nitrate	Rep2 Nitrate	Nitrate absolute difference	Rep1 Phosphate	Rep2 Phosphate	Phosphate absolute difference	Rep1 Nitrite	Rep2 Nitrite	Nitrite absolute difference
BIO	0.25	0.26	**0.01**	1.41	1.41	**0.01**	0.10	0.10	**0.00**
BIO	32.01	31.43	**0.58**	1.40	1.29	**0.11**	0.54	0.55	**0.01**
BIO	2.55	2.52	**0.04**	1.08	1.15	**0.08**	0.28	0.29	**0.01**
BIO	0.23	0.23	**0.00**	0.73	0.70	**0.03**	0.11	0.12	**0.01**
BIO	1.08	0.77	**0.31**	0.67	0.65	**0.02**	0.16	0.15	**0.01**
BIO	1.21	1.24	**0.03**	0.91	0.95	**0.04**	0.18	0.18	**0.00**
BIO	0.61	0.38	**0.23**	0.90	0.89	**0.01**	0.12	0.10	**0.02**
BIO	2.16	2.16	**0.00**	0.49	0.52	**0.03**	0.25	0.25	**0.00**
BIO	31.94	31.89	**0.04**	0.99	0.99	**0.00**	0.51	0.52	**0.01**
BIO	0.29	0.23	**0.06**	0.25	0.19	**0.07**	0.09	0.08	**0.01**
BIO	0.76	0.72	**0.04**	0.26	0.24	**0.02**	0.12	0.13	**0.01**
BIO	1.20	1.22	**0.01**	0.72	0.75	**0.04**	0.16	0.16	**0.00**
BIO	0.55	0.54	**0.01**	0.67	0.67	**0.00**	0.10	0.10	**0.00**
BIO	0.26	0.24	**0.02**	0.25	0.25	**0.00**	0.14	0.14	**0.00**
BIO	0.25	0.30	**0.05**	0.55	0.57	**0.02**	0.14	0.16	**0.02**
BIO	25.74	27.57	**1.83**	0.76	0.74	**0.03**	1.04	1.12	**0.08**
BIO	11.94	10.42	**1.52**	0.78	0.89	**0.11**	0.89	0.85	**0.05**
BIO	6.22	5.27	**0.95**	1.15	1.16	**0.01**	0.65	0.60	**0.05**
BIO	35.46	35.45	**0.00**	2.20	2.50	**0.30**	2.38	2.69	**0.31**
BIO	0.72	0.39	**0.33**	0.49	0.46	**0.03**	0.16	0.14	**0.02**
BIO	0.35	0.33	**0.03**	0.27	0.26	**0.01**	0.13	0.13	**0.00**
BIO	24.76	24.80	**0.04**	0.50	0.49	**0.01**	1.05	1.05	**0.00**
BIO	11.04	10.94	**0.10**	0.39	0.38	**0.01**	0.90	0.91	**0.01**
BIO	6.20	6.17	**0.03**	0.62	0.66	**0.04**	0.62	0.62	**0.00**
BIO	35.40	35.40	**0.00**	2.17	2.14	**0.03**	2.68	2.67	**0.00**
BIO	0.90	0.42	**0.48**	8.43	9.73	**1.30**	0.16	0.15	**0.00**
BIO	0.42	0.39	**0.03**	1.06	1.03	**0.03**	0.16	0.16	**0.00**

Laboratory	Rep1 Nitrate	Rep2 Nitrate	Nitrate absolute difference	Rep1 Phosphate	Rep2 Phosphate	Phosphate absolute difference	Rep1 Nitrite	Rep2 Nitrite	Nitrite absolute difference
BIO	0.36	0.31	**0.05**	0.93	0.91	**0.02**	0.16	0.14	**0.02**
BIO	0.53	0.37	**0.17**	0.74	0.73	**0.01**	0.18	0.14	**0.04**
BIO	0.37	0.31	**0.06**	0.86	0.82	**0.04**	0.11	0.11	**0.00**
BIO	0.34	0.34	**0.00**	0.97	0.96	**0.00**	0.14	0.14	**0.00**
BIO	0.32	0.31	**0.02**	0.89	0.88	**0.02**	0.11	0.11	**0.00**
BIO	0.31	0.31	**0.00**	0.66	0.64	**0.02**	0.12	0.11	**0.00**
BIO	0.46	0.43	**0.03**	1.02	1.02	**0.00**	0.16	0.17	**0.00**
BIO	0.36	0.33	**0.03**	1.27	1.17	**0.10**	0.21	0.21	**0.00**
BIO	1.31	1.49	**0.19**	1.38	1.39	**0.02**	0.41	0.42	**0.01**
BIO	0.34	0.32	**0.02**	0.85	0.94	**0.08**	0.16	0.15	**0.01**
BIO	0.32	0.36	**0.04**	0.86	0.86	**0.00**	0.16	0.15	**0.01**
BIO	0.39	0.39	**0.00**	0.84	0.82	**0.02**	0.15	0.15	**0.01**
BIO	0.31	0.32	**0.01**	0.61	0.63	**0.02**	0.14	0.14	**0.00**
BIO	1.37	1.39	**0.03**	1.19	1.22	**0.03**	0.38	0.39	**0.01**
BIO	0.32	0.32	**0.00**	0.71	0.69	**0.01**	0.12	0.12	**0.00**
BIO	0.51	0.53	**0.02**	0.68	0.70	**0.02**	0.37	0.40	**0.03**
BIO	0.34	0.33	**0.01**	0.71	0.71	**0.00**	0.14	0.12	**0.02**
MLI	1.13	0.97	**0.16**	0.42	0.61	**0.19**	0.18	0.17	**0.01**
MLI	2.19	2.20	**0.01**	0.41	0.54	**0.13**	0.15	0.17	**0.02**
MLI	0.29	0.22	**0.07**	0.37	0.32	**0.05**	0.11	0.09	**0.02**
MLI	2.88	3.76	**0.88**	31.94	29.12	**2.82**	2.28	2.03	**0.25**
MLI	1.36	1.49	**0.13**	1.51	1.57	**0.06**	0.30	0.34	**0.04**
MLI	0.72	0.63	**0.09**	0.75	0.66	**0.09**	0.18	0.16	**0.01**
MLI	0.54	0.48	**0.06**	0.45	0.41	**0.04**	0.09	0.08	**0.01**
MLI	2.89	2.80	**0.09**	0.30	0.24	**0.06**	0.13	0.12	**0.01**
MLI	0.21	0.14	**0.07**	0.77	1.02	**0.25**	0.14	0.14	**0.00**
MLI	2.01	1.90	**0.11**	31.80	32.47	**0.67**	0.51	0.50	**0.01**

Laboratory	Rep1 Nitrate	Rep2 Nitrate	Nitrate absolute difference	Rep1 Phosphate	Rep2 Phosphate	Phosphate absolute difference	Rep1 Nitrite	Rep2 Nitrite	Nitrite absolute difference
MLI	1.55	1.41	**0.14**	0.94	0.89	**0.05**	0.26	0.21	**0.05**
MLI	0.60	0.51	**0.09**	0.58	0.30	**0.28**	0.12	0.11	**0.01**
MLI	0.23	0.23	**0.00**	1.21	1.11	**0.10**	0.09	0.09	**0.00**
MLI	0.27	0.33	**0.06**	1.54	2.34	**0.80**	0.08	0.10	**0.02**
MLI	1.30	1.38	**0.08**	1.97	2.28	**0.31**	0.13	0.15	**0.01**
MLI	10.35	10.46	**0.11**	3.66	4.02	**0.36**	6.59	6.89	**0.30**
MLI	0.46	0.55	**0.09**	1.38	2.09	**0.71**	0.19	0.21	**0.02**
MLI	0.16	0.17	**0.01**	1.57	1.94	**0.37**	0.07	0.11	**0.04**
MLI	0.22	0.17	**0.05**	0.98	1.04	**0.06**	0.07	0.06	**0.02**
MLI	0.28	0.21	**0.07**	1.40	1.42	**0.02**	0.06	0.04	**0.02**
MLI	1.19	1.14	**0.05**	1.64	1.46	**0.18**	0.10	0.09	**0.01**
MLI	9.47	8.43	**1.04**	2.64	2.30	**0.34**	6.36	5.64	**0.72**
MLI	0.42	0.33	**0.09**	1.11	0.97	**0.14**	0.12	0.10	**0.02**
MLI	0.19	0.15	**0.04**	1.25	1.23	**0.02**	0.05	0.06	**0.01**
MLI	2.49	3.43	**0.94**	0.87	1.24	**0.37**	0.23	0.31	**0.08**
MLI	37.51	36.11	**1.40**	1.07	1.16	**0.09**	0.66	0.60	**0.05**
MLI	0.26	0.11	**0.15**	0.63	0.50	**0.13**	0.11	0.09	**0.02**
MLI	1.59	0.96	**0.63**	0.39	0.43	**0.04**	0.14	0.13	**0.02**
MLI	1.59	2.03	**0.44**	0.69	0.81	**0.12**	0.15	0.17	**0.02**
MLI	0.69	0.97	**0.28**	0.85	0.71	**0.14**	0.09	0.10	**0.01**
MLI	2.29	2.72	**0.43**	0.33	0.36	**0.03**	0.18	0.21	**0.03**
MLI	24.75	28.68	**3.93**	0.90	0.94	**0.04**	0.47	0.49	**0.02**
MLI	0.10	0.09	**0.01**	0.13	0.04	**0.09**	0.02	0.02	**0.00**
MLI	1.33	0.81	**0.52**	0.51	0.12	**0.39**	0.11	0.07	**0.05**
MLI	1.53	1.61	**0.08**	0.66	0.59	**0.07**	0.11	0.11	**0.00**
MLI	0.92	0.67	**0.25**	1.06	0.62	**0.44**	0.07	0.05	**0.01**
MLI	0.16	0.15	**0.01**	0.21	0.18	**0.03**	0.11	0.12	**0.01**

Laboratory	Rep1 Nitrate	Rep2 Nitrate	Nitrate absolute difference	Rep1 Phosphate	Rep2 Phosphate	Phosphate absolute difference	Rep1 Nitrite	Rep2 Nitrite	Nitrite absolute difference
MLI	0.17	0.10	**0.07**	0.50	0.44	**0.06**	0.15	0.14	**0.01**
MLI	36.34	36.03	**0.31**	0.87	0.63	**0.24**	1.29	1.21	**0.07**
MLI	18.54	18.30	**0.24**	0.84	0.58	**0.26**	1.16	1.11	**0.04**
MLI	7.47	6.80	**0.67**	0.92	0.82	**0.10**	0.58	0.66	**0.08**
MLI	161.81	162.45	**0.64**	3.38	2.61	**0.77**	2.99	2.85	**0.14**
MLI	0.17	0.13	**0.04**	0.09	0.10	**0.01**	0.03	0.04	**0.01**
MLI	0.19	0.10	**0.09**	0.20	0.05	**0.15**	0.04	0.05	**0.00**
MLI	26.58	25.95	**0.63**	0.38	0.16	**0.22**	0.94	0.89	**0.05**
MLI	11.82	11.58	**0.24**	0.29	0.11	**0.18**	0.78	0.74	**0.04**
MLI	6.70	6.51	**0.19**	0.51	0.27	**0.24**	0.51	0.47	**0.04**
MLI	178.03	177.32	**0.70**	2.24	1.77	**0.47**	3.07	3.04	**0.03**
MLI	0.12	0.13	**0.01**	0.66	0.91	**0.25**	0.06	0.07	**0.01**
MLI	0.18	0.18	**0.00**	0.84	1.15	**0.31**	0.08	0.09	**0.01**
MLI	0.12	0.09	**0.03**	0.77	0.69	**0.08**	0.07	0.05	**0.01**
MLI	0.13	0.08	**0.05**	0.56	0.51	**0.05**	0.05	0.06	**0.00**
MLI	0.10	0.08	**0.02**	0.62	0.59	**0.03**	0.02	0.04	**0.02**
MLI	0.17	0.12	**0.05**	0.74	0.77	**0.03**	0.05	0.05	**0.00**
MLI	0.12	0.09	**0.03**	0.69	0.65	**0.04**	0.02	0.03	**0.01**
MLI	0.14	0.13	**0.01**	0.48	0.46	**0.02**	0.02	0.06	**0.04**
MLI	0.26	0.23	**0.03**	0.82	0.66	**0.16**	0.10	0.10	**0.01**
MLI	0.17	0.12	**0.05**	1.25	0.97	**0.28**	0.19	0.14	**0.05**
MLI	1.31	1.39	**0.08**	1.01	1.02	**0.01**	0.27	0.29	**0.02**
MLI	0.13	0.12	**0.01**	0.61	0.77	**0.16**	0.07	0.08	**0.01**
MLI	0.69	0.51	**0.18**	2.09	1.56	**0.53**	1.49	1.03	**0.46**
MLI	0.18	0.11	**0.07**	0.61	0.51	**0.10**	0.10	0.07	**0.03**
MLI	0.27	0.18	**0.09**	0.67	0.47	**0.20**	0.09	0.05	**0.03**
MLI	0.15	0.09	**0.06**	0.50	0.42	**0.08**	0.06	0.04	**0.02**

Laboratory	Rep1 Nitrate	Rep2 Nitrate	Nitrate absolute difference	Rep1 Phosphate	Rep2 Phosphate	Phosphate absolute difference	Rep1 Nitrite	Rep2 Nitrite	Nitrite absolute difference
MLI	1.52	1.39	**0.13**	0.98	0.79	**0.19**	0.28	0.24	**0.04**
MLI	0.14	0.07	**0.07**	0.54	0.45	**0.09**	0.04	0.03	**0.01**
MLI	0.35	0.27	**0.08**	0.60	0.50	**0.10**	0.26	0.25	**0.02**
MLI	0.16	0.09	**0.07**	0.55	0.52	**0.03**	0.05	0.04	**0.01**
		Average	**0.22**			**0.56**			**0.03**
		variance	**0.31**			**10.19**			**0.01**
		Min	**0.00**			**0.00**			**0.00**
		Max	**4.20**			**27.80**			**0.72**

Appendix 2: Water sample results from the 2009 calibration exercise for CAMP nutrient sampling. Data from duplicate water samples collected at each station were averaged to produce a best estimate of nitrate, nitrite and phosphate (phos.) at each station. Appendix 2 gives calculated values for the percent differences (% diff)* between Bedford Institute of Oceanography (BIO) and Maurice Lamontagne Institute (MLI) and between filtered (F) and unfiltered (UF) water samples. All data are in µM.
Legend: SCOU = Scoudouc River, BOUC = Bouctouche, TROU = Trout River, SUMM = Summerside, PUGW = Pugwash, PICT = Pictou.* % difference = {1-(smallest value/largest value)}x100

Nitrate Table

Block	Nitrate F BIO	Nitrate F IML	Nitrate UF BIO	Nitrate UF IML	% diff btw BIO and IML for F	% diff btw BIO and IML for UF	% diff btw F and UF for BIO	% diff btw F and UF for IML
SCOU 1	0.520	0.510	0.997	1.050	1.923	5.095	47.817	51.429
SCOU 2	2.399	2.845	2.263	2.195	15.677	3.005	5.669	22.847
SCOU 3	0.327	0.175	0.284	0.255	46.483	10.211	13.150	31.373
SCOU 4	1.573	1.955	2.035	3.320	19.540	38.705	22.703	41.114
SCOU 5	1.213	1.480	1.249	1.425	18.041	12.386	2.843	3.716
SCOU 6	0.540	0.555	0.661	0.675	2.793	2.074	18.381	17.778
BOUC 1	0.239	0.195	0.274	0.230	18.410	16.058	12.774	15.217
BOUC 2	0.252	0.245	0.267	0.300	2.584	11.167	5.629	18.333
BOUC 3	0.939	1.165	0.798	1.340	19.399	40.448	15.016	13.060
BOUC 4	10.284	8.950	18.561	10.402	12.967	43.958	44.595	13.957
BOUC 5	0.384	0.375	0.416	0.505	2.216	17.624	7.813	25.743
BOUC 6	0.252	0.170	0.227	0.165	32.406	27.313	9.742	2.941
TROU 1	2.161	2.505	2.535	2.960	13.752	14.375	14.756	15.372
TROU 2	31.913	26.715	31.717	36.810	16.286	13.836	0.613	27.424
TROU 3	0.264	0.095	0.229	0.185	63.947	19.037	13.283	48.649
TROU 4	0.741	1.070	0.921	1.275	30.794	27.765	19.598	16.078
TROU 5	1.209	1.570	1.227	1.810	22.994	32.210	1.467	13.260
TROU 6	0.548	0.795	0.494	0.830	31.132	40.542	9.863	4.217
SUMM 1	0.553	0.150	0.249	0.155	72.875	37.626	55.063	3.226
SUMM 2	0.341	0.145	0.277	0.135	57.416	51.175	18.796	6.897
SUMM 3	24.777	26.265	26.651	36.185	5.665	26.349	7.030	27.415
SUMM 4	10.989	11.700	11.182	18.420	6.077	39.297	1.722	36.482
SUMM 5	6.184	6.605	5.745	7.135	6.374	19.481	7.099	7.428
SUMM 6	35.401	177.674	35.460	162.134	80.075	78.129	0.166	8.746

Block	Nitrate F BIO	Nitrate F IML	Nitrate UF BIO	Nitrate UF IML	% diff btw BIO and IML for F	% diff btw BIO and IML for UF	% diff btw F and UF for BIO	% diff btw F and UF for IML
PUGW 5	0.336	0.090	0.661	0.125	73.174	81.075	49.205	28.000
PUGW 4	0.341	0.145	0.406	0.180	57.478	55.665	16.010	19.444
PUGW 3	0.316	0.105	0.338	0.105	66.719	68.889	6.519	0.000
PUGW 6	0.308	0.135	0.448	0.105	56.169	76.563	31.250	22.222
PICT 1	0.387	0.225	0.445	0.245	41.860	44.944	13.034	8.163
PICT 2	0.314	0.120	0.343	0.145	61.722	57.726	8.601	17.241
PICT 3	1.379	1.455	1.398	1.350	5.258	3.399	1.360	7.216
PICT 4	0.321	0.105	0.333	0.125	67.239	62.406	3.609	16.000
PICT 5	0.517	0.310		0.600	40.039			48.333
PICT 6	0.338	0.125	0.338	0.145	63.018	57.101	0.000	13.793
Average	**4.075**	**8.139**	**4.528**	**8.618**	**33.309**	**34.413**	**14.702**	**19.209**
Min	**0.239**	**0.090**	**0.227**	**0.105**	**1.923**	**2.074**	**0.000**	**0.000**
Max	**35.401**	**177.674**	**35.460**	**162.134**	**80.075**	**81.075**	**55.063**	**51.429**

Nitrite Table

Block	Nitrite F BIO	Nitrite F IML	Nitrite UF BIO	Nitrite UF IML	% diff btw BIO and IML for F	% diff btw BIO and IML for UF	% diff btw F and UF for BIO	% diff btw F and UF for IML
SCOU 1	0.166	0.083	0.213	0.173	50.000	19.014	22.066	51.884
SCOU 2	0.195	0.128	0.238	0.164	34.359	31.303	18.067	21.713
SCOU 3	0.158	0.138	0.149	0.097	12.698	35.235	5.397	29.818
SCOU 4	0.360	0.507	0.686	2.158	28.994	68.204	47.522	76.501
SCOU 5	0.310	0.233	0.361	0.318	24.839	11.911	14.127	26.730
SCOU 6	0.193	0.115	0.241	0.170	40.415	29.461	19.917	32.353
BOUC 1	0.113	0.065	0.141	0.087	42.222	38.652	20.213	24.855
BOUC 2	0.106	0.053	0.121	0.091	50.237	24.481	12.448	42.308
BOUC 3	0.157	0.092	0.167	0.141	41.720	15.868	5.988	34.875
BOUC 4	2.608	6.001	2.616	6.743	56.549	61.203	0.325	11.002
BOUC 5	0.171	0.109	0.211	0.201	36.070	4.513	19.002	45.771
BOUC 6	0.101	0.056	0.108	0.091	45.050	15.814	6.047	38.674
TROU 1	0.249	0.194	0.284	0.272	21.932	4.225	12.500	28.676
TROU 2	0.515	0.479	0.548	0.630	6.888	13.016	6.113	23.959
TROU 3	0.084	0.017	0.113	0.103	80.357	8.850	25.664	83.981

Block	Nitrite F BIO	Nitrite F IML	Nitrite UF BIO	Nitrite UF IML	% diff btw BIO and IML for F	% diff btw BIO and IML for UF	% diff btw F and UF for BIO	% diff btw F and UF for IML
TROU 4	0.125	0.090	0.154	0.134	28.400	12.987	18.831	33.209
TROU 5	0.162	0.107	0.182	0.158	33.951	12.948	10.744	32.278
TROU 6	0.098	0.060	0.111	0.092	38.974	16.742	11.765	35.326
SUMM 1	0.148	0.035	0.136	0.110	76.689	19.118	99.868	68.636
SUMM 2	0.128	0.044	0.152	0.149	65.625	1.974	15.789	70.470
SUMM 3	1.054	0.912	1.080	1.250	13.431	13.565	2.454	27.011
SUMM 4	0.905	0.763	0.870	1.137	15.746	23.449	3.867	32.908
SUMM 5	0.622	0.492	0.627	0.618	20.837	1.437	0.798	20.324
SUMM 6	2.675	3.057	2.533	2.920	12.519	13.256	5.309	4.505
PUGW 5	0.113	0.030	0.153	0.064	73.894	58.497	26.144	53.543
PUGW 4	0.137	0.051	0.159	0.087	63.004	45.283	14.151	41.954
PUGW 3	0.111	0.028	0.149	0.060	74.661	59.933	25.589	52.941
PUGW 6	0.114	0.042	0.158	0.056	63.436	64.444	27.937	25.893
PICT 1	0.150	0.071	0.166	0.101	52.667	39.458	9.639	29.353
PICT 2	0.138	0.054	0.208	0.165	61.091	20.673	33.894	67.576
PICT 3	0.382	0.264	0.413	0.281	30.931	31.879	7.515	6.228
PICT 4	0.121	0.034	0.154	0.072	72.314	53.420	21.173	53.147
PICT 5	0.382	0.257		1.257	32.765			79.586
PICT 6	0.129	0.042	0.156	0.084	67.315	45.981	17.363	50.000
Average	**0.387**	**0.432**	**0.417**	**0.595**	**43.252**	**27.782**	**17.825**	**39.941**
Min	**0.084**	**0.017**	**0.108**	**0.056**	**6.888**	**1.437**	**0.325**	**4.505**
Max	**2.675**	**6.001**	**2.616**	**6.743**	**80.357**	**68.204**	**99.868**	**83.981**

Phosphate Table

Block	Phos. F BIO	Phos. F IML	Phos. UF BIO	Phos. UF IML	% diff btw BIO and IML for F	% diff btw BIO and IML for UF	% diff btw F and UF for BIO	% diff btw F and UF for IML
SCOU 1	0.623	0.430	0.635	0.515	30.924	18.898	1.969	16.505
SCOU 2	0.444	0.270	0.960	0.475	39.189	50.521	53.750	43.158
SCOU 3	0.533	0.895	0.899	0.345	98.842	61.624	40.768	61.453
SCOU 4	85.100	32.137	30.800	30.528	62.236	0.883	63.807	5.007
SCOU 5	1.243	0.915	1.433	1.540	26.358	6.981	13.264	40.584
SCOU 6	0.649	0.440	0.879	0.705	32.203	19.750	26.124	37.589
BOUC 1	1.156	1.010	1.487	1.160	12.592	21.991	22.293	12.931
BOUC 2	1.559	1.410	1.769	1.940	9.528	8.814	11.899	27.320
BOUC 3	1.739	1.550	1.886	2.125	10.868	11.247	7.794	27.059
BOUC 4	36.600	2.470	62.600	3.840	93.251	93.866	41.534	35.677
BOUC 5	1.271	1.040	1.326	1.735	18.142	23.602	4.149	40.058
BOUC 6	1.410	1.240	1.376	1.755	12.057	21.595	2.411	29.345
TROU 1	0.501	0.345	1.113	1.055	31.138	5.211	54.987	67.299
TROU 2	0.986	0.918	1.347	1.115	6.829	17.223	26.837	17.650
TROU 3	0.219	0.085	0.717	0.565	61.187	21.144	69.435	84.956
TROU 4	0.250	0.315	0.664	0.410	20.794	38.206	62.396	23.171
TROU 5	0.736	0.625	0.927	0.750	15.024	19.094	20.658	16.667
TROU 6	0.668	0.840	0.898	0.780	20.476	13.092	25.571	7.143
SUMM 1	0.475	0.095	0.253	0.195	80.000	22.925	46.737	51.282
SUMM 2	0.262	0.125	0.558	0.470	52.290	15.695	53.004	73.404
SUMM 3	0.496	0.270	0.751	0.750	45.565	0.067	33.911	64.000
SUMM 4	0.385	0.200	0.834	0.710	48.052	14.868	53.837	71.831
SUMM 5	0.641	0.390	1.156	0.870	39.158	24.740	44.550	55.172
SUMM 6	2.155	2.005	2.350	2.995	6.961	21.536	8.298	33.055
PUGW 5	0.835	0.605	9.080	0.785	27.545	91.355	90.804	22.930
PUGW 4	0.965	0.755	1.045	0.995	21.762	4.785	7.656	24.121
PUGW 3	0.885	0.670	0.924	0.730	24.251	20.953	4.223	8.219
PUGW 6	0.652	0.470	0.730	0.535	27.859	26.712	10.753	12.150
PICT 1	0.828	0.570	1.021	0.740	31.118	27.487	18.912	22.973
PICT 2	0.618	0.460	1.219	1.110	25.566	8.942	49.303	58.559
PICT 3	1.209	0.885	1.386	1.015	26.769	26.741	12.775	12.808
PICT 4	0.700	0.495	0.893	0.690	29.286	22.732	21.613	28.261

Block	Phos. F BIO	Phos. F IML	Phos. UF BIO	Phos. UF IML	% diff btw BIO and IML for F	% diff btw BIO and IML for UF	% diff btw F and UF for BIO	% diff btw F and UF for IML
PICT 5	0.692	0.550		1.825	20.463			69.863
PICT 6	0.706	0.535	0.859	0.560	24.167	34.808	17.870	4.464
Average	**4.358**	**1.648**	**4.084**	**1.950**	**33.307**	**24.791**	**31.027**	**35.490**
Min	**0.219**	**0.085**	**0.253**	**0.195**	**6.829**	**0.067**	**1.969**	**4.464**
Max	**85.100**	**32.137**	**62.600**	**30.528**	**98.842**	**93.866**	**90.804**	**84.956**