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FINAL REPORT

CHESAPEAKE BAY PROGRAM BLIND AUDIT

Fiscal Year 2007 Final Report

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INTRODUCTION

The purpose of this Blind Audit Program is to provide samples of specific nutrient analytes at concentrations commonly found in estuarine systems for analysis by laboratories that analyze water samples collected from the Chesapeake Bay and its tributaries. The concentrations of these samples, which are unknown to the recipient analysts, are compared to their prepared concentrations.

In the early years of the Chesapeake Bay Program, U.S. EPA provided blind audit samples on an irregular basis to laboratories analyzing Chesapeake Bay water samples. However, these audit samples were designed for waste water/drinking water applications rather than for estuarine water applications. Consequently, the concentrations were much higher than normally occur in the Bay and did not provide a reasonable estimate of accuracy for low level nutrient concentrations. For example, a blind audit concentration of 1.0 mg NH4-N/L would be comparable to NPDES water samples, but would be at least an order of magnitude greater than concentrations normally occurring in most parts of Chesapeake Bay.

The only continuous program providing an estimate of laboratory performance has been the Chesapeake Bay Coordinated Split Sample Program (CSSP). Data generated from this program provide the only long term QA/QC data base to compare nutrient measurements provided by laboratories analyzing water samples collected from Chesapeake Bay and its tributaries. Samples for CSSP are natural water samples collected from Chesapeake Bay or a tributary. Briefly, a common unfiltered water sample is distributed to the various field/laboratory personnel who, in turn, subsample into dissolved and particulate fractions. These are analyzed and the results compared to those of other participating laboratories. Resulting data analysis can show how field filtration techniques and/or laboratory practices affect data variability. CSSP samples are each subject to cumulative errors of analytical determinations from variation in both field and laboratory procedures. Also, these data sets cannot definitively determine the accuracy of laboratory analyses.

The current Blind Audit Program has been designed to complement the CSSP. Blind Audit particulate samples distributed to participants have few cumulative errors associated with field filtering and subsampling procedures. Prepared concentrates of dissolved substances, whose concentrations are unknown to the analysts, are provided so that laboratory accuracy can be assessed.

This is the ninth year of the Blind Audit Program and it is the continued intent of this program to provide unknown, low level dissolved and particulate nutrient samples to laboratories analyzing Chesapeake Bay Program nutrients, as well as to other laboratories interested in participating in the Blind Audit Program.

MATERIALS AND METHODS

Blind Audit samples were sent to participating laboratories on 22 August 2006 and 12 February 2007. Participating laboratories and contact personnel are found in Table 1.

Parameters measured were: total dissolved nitrogen (organic N), total dissolved phosphorus (organic P), nitrate+nitrite, ammonium, phosphate and dissolved organic carbon. High and low concentration samples were provided for each analyte. Particulate carbon, nitrogen and phosphorus, chlorophyll and total suspended solids, were also provided for those laboratories that routinely analyze these parameters. Chlorophyll samples were natural population samples

collected from the mouth of the Patuxent River.

Dissolved Blind Audit concentrates were prepared by careful dilution of high quality standards using 18.3 megohm deionized water. The concentrates were sealed in 20 mL ampoules for shipment to participants. One ampoule contained a concentrate of an organic nitrogen compound and an organic phosphorus compound to be diluted for the analysis of low level total dissolved nitrogen and total dissolved phosphorus. A second ampoule contained a concentrate of an organic nitrogen compound and an organic phosphorus compound to be diluted for the analysis of higher level total dissolved nitrogen and total dissolved phosphorus. A third ampoule contained a concentrate to be diluted for the analysis of low level inorganic nutrients (ammonium, nitrate and phosphate). A fourth ampoule contained a concentrate to be diluted for the analysis of higher level inorganic nutrients. The fifth and sixth ampoules contained a low and high concentration of dissolved organic carbon (Potassium hydrogen phthalate), respectively. At each participating laboratory, an aliquot from each ampoule was diluted and analyzed according to accompanying instructions for preparation and dilution. These Blind Audit samples were then inserted randomly in a typical estuarine sample set. Final concentrations were reported for each diluted concentrate according to the dilution instructions provided.

Particulate analytes are measured by analyzing suspended material concentrated on filter pads. There are no commercially available suspensions of pure carbon, nitrogen or phosphorus compounds, so a natural sample was subsampled onto filter pads for analysis by participating laboratories. A batch water sample was collected from the CBL pier, and subsampled for particulate samples of carbon, nitrogen and phosphorus. Particulate C/N samples were filtered from the batch sample with care taken to shake the batch before each filtration to ensure homogeneity. Vacuum filtration was used to process the filters. Samples were dried completely (overnight at -47EC) before shipment. Two samples on 25 mm GF/F pads were sent to each laboratory for analysis.

The same general procedure was followed for particulate phosphorus samples in which they were concentrated by vacuum filtration on 47 mm GF/F pads.

Filter pads were sent to each laboratory for the analysis of particulate C, N, and P. The volume of sample filtered was noted in the instructions so that each laboratory could report concentrations in mg/L. Samples for chlorophyll analysis were filtered from natural population samples onto 47 mm GF/F filter pads. Replicate pads were provided to participating laboratories.

A suspension of a known mass of infusorial earth in deionized water was stirred with a magnetic stirrer. While stirring continued, an aliquot was subsampled by pipette into a screw cap vial for each participating laboratory. Detailed instructions explaining how to prepare this concentrate for total suspended solids analysis, were also provided.

Samples were sent in coolers via next day carrier to the participating laboratories. A cold temperature was required for chlorophyll samples, so frozen cold packs were packed in those participants= coolers.

RESULTS

Tables and figures summarizing results from the summer 2006 and winter 2007 audit are found at the end of the report. Shortly after the completion of the study, a brief data report, including the concentrations of the prepared samples, was sent to each participant for them to check their data. These data reviews served as a final check of data before preparing this final report.

Concentrations were assessed statistically by calculating the mean and standard deviation of each sample set, then calculating how many standard deviations separated each laboratory=s reported concentration from that mean (Table 2). The percent recovery of each laboratory=s reported concentration relative to the prepared concentration was also calculated for the dissolved analytes (Table 3 and Appendix 1).

DISSOLVED FRACTION

Total Dissolved Nitrogen: With the exception of one participant's result for the low concentration from the winter 2007 audit, results for all the audits had approximately the same agreement with the prepared concentration and between the participants.

Total Dissolved Phosphorus: With the exception of one participant's result for the high concentration from the winter 2007 audit, results for all the audits had approximately the same agreement with the prepared concentration and between the participants. The variation between the reported and prepared concentrations was generally less than in the past 2 years. However, for the high concentration winter 2007 audit, one laboratory's result was approximately double those of the other participants.

Ammonium: Results for the high concentration ammonium summer 2006 audit had close agreement with the prepared concentration and between the participants. The prepared high concentration for the winter 2007 audit was approximately one third that of the summer 2006 audit. Results for the high concentration ammonium winter 2007 audit had less agreement with the prepared concentration and between the participants. The variation of the data reported by participants for the low level ammonium summer 2006 audit was quite large, i.e., the proportion of the standard deviation to the mean for the low level ammonium sample was quite large. The variation between the reported and prepared concentration for the low level ammonium winter 2007 audit was generally less than in the past 3 years.

Nitrate + Nitrite: For the prepared high level concentrations of nitrate + nitrite, most participants reported approximately the same concentration. For the low level nitrate + nitrite concentration, there was slightly more variability between participants and from the prepared concentration. For the high level concentration summer 2006 audit, all reported concentrations were within 10% of the prepared concentration.

Orthophosphate: For the prepared high level concentrations of orthophosphate, most participants reported approximately the same concentration. For the low level orthophosphate concentration, there was considerable variability between participants and from the prepared concentration.

Dissolved Organic Carbon: Results for both audits had approximately the same agreement with the prepared concentration and between the participants. For the low level dissolved organic carbon concentration, there was more variability between participants and from the prepared concentration. For the high level concentration winter 2007 audit, all reported concentrations

were within 10% of the prepared concentration.

PARTICULATE FRACTION

Again, it should be noted that particulate carbon, nitrogen and phosphorus samples were filtered from a common estuarine water sample and, consequently, are not true blind audit samples produced from pure constituents. Particulate results are graphically presented in Figures 1 and 5.

Particulate Nitrogen: Particulate N results for the summer 2006 audit revealed close agreement between all of the participating laboratories (Table 2). As in past years, this was remarkably close agreement for comparison of samples of a natural population by multiple laboratories. For the winter 2007 audit, one laboratory's result was approximately double those of the other participants.

Particulate Carbon: Particulate C results for both audits revealed close agreement between all participating laboratories (Table 2). Again, this is remarkably close agreement for multi-laboratory comparison of samples of a natural population!

Particulate Phosphorus: Particulate P results for both audits revealed very close agreement between all of the participating laboratories (Table 2). As in past years, this was remarkably close agreement for comparison of samples of a natural population by multiple laboratories.

Chlorophyll: Chlorophyll results for both audits displayed the usual close agreement that was remarkable for multi-laboratory comparison of such low concentrations of an environmentally transitory compound. One laboratory's data for the winter 2007 audit were considerably different from all other participants' data.

Total Suspended Solids: The concentrate of infusorial earth suspended in deionized water was suspended further in deionized water by each laboratory, then concentrated on a filter pad and weighed. For the summer 2006 sample, 15.0 mg/L was prepared, and there was a consistent slight negative bias reported by most participants. For the winter 2007 sample, 13.0 mg/L was prepared but, there was, again, a consistent negative bias reported by most participants, as well as greater variability between the results reported by the participants than in any previous audits.

DISCUSSION

Several important issues should be considered when assessing whether individual Blind Audit results are within acceptable limits.

Variation Associated With An Analytical Method: As we have noted in previous Blind Audit Reports, analytical variability is associated with any quantitative determination. The method detection limit (three times the standard deviation of seven low level replicate natural samples) is often used to express that level of variation. Total dissolved nitrogen data provide a good example. The detection limit at CBL has been determined to be 0.02 mg N/L. Any total dissolved nitrogen measurement has a potential 0.02 mg N/L variability associated with it. This variability, when expressed as a percent of the Atrue@ concentration, can be extremely large for low level concentrations and fairly low for higher concentrations. For example, a 0.20 mg N/L concentration has an analytical variability of 10% associated with it; whereas, a 1.20 mg N/L concentration has an analytical variability of 2%.

Acceptance Limits of Provided Dissolved Samples: Companies that prepare large quantities of performance evaluation samples assign acceptable confidence limits around the Atrue@ value. In one case (SPEX, CertiPrep), the mean recovery and standard deviation are later reported along with the true concentration and the 95% confidence interval (CI). The 95% CI is the mean recovery ∀ 2 standard deviations and is developed from regression equations from Water Pollution Performance Evaluation Studies. A recently purchased set of these standards gave a true total P value of 3.00 mg P/L with a 95% CI of 2.47-3.42 mg P/L. The lower end of the 95% CI recovery allows 82% recovery of the true concentration. This type of statistical analysis was not performed on the Blind Audit Program samples prepared for this study prior to their distribution to the participants.

Parameters assessed in the Blind Audit do not have predetermined acceptance limits, so we are following the statistical procedure of ERA, an approved source of wastewater and drinking water proficiency samples, and the State of Wisconsin Proficiency Testing program. They average the results for each parameter and at each concentration, then calculate the standard deviation from the mean. Results that are within 2 standard deviations Apass@, and those greater than 3 standard deviations Afail@. Results between 2 and 3 standard deviations are in the Awarning@ category.

Most of the data comparisons based on standard deviations showed similar characteristics (Table 2); that is, the reported concentrations were similar, and one or two concentrations fell slightly beyond one standard deviation from the mean of all data for that portion of the study. Apparently, it is a statistical Areality@ in small sample sets with little variability between individual points, that at least one point will lie just beyond one standard deviation from the mean. Thus, for most of the data sets compared by means and standard deviations, all the reported concentrations Apassed.@ It should also be noted that approximately the same number were in the Awarning@ category as in most of the previous studies, and that two data points fell in the Afail@ category in each audit.

Data sets with relatively small standard deviations yielded more Awarning@ points. For example, in the summer 2006 blind audit of high level nitrate+nitrite concentration, the mean reported concentration was 0.717 mg N/L and reported concentrations ranged from 0.697-.762 mg N/L (Coefficient of Variation, 2.9%). Eleven laboratories reported results for this high level sample that were within two standard deviations (S.D. ∀ 0.0207 mg N/L) of the mean. Since the standard deviation was so small, one laboratory's reported result for this sample was between two and three standard deviations of the mean, so was labeled Awarning,@ although <u>all</u> of the reported data were within ∀5% of the prepared concentration. Thus, by that measure of accuracy, all of the data Apassed.@ This nitrate+nitrite data comparison points toward a form of circular reasoning in these statistical assessments. The data being evaluated are also the data that were used to calculate the mean and standard deviation to which the data are being compared.

Data were also assessed by comparing reported concentrations to those that had been prepared (Table 3). Groupings of data in Apass, warn and fail@ categories were arbitrarily set. Reported data that were within ∀10% of the prepared concentration were listed as Apass.@ Reported data that were 80-90% or 110 -120% of the prepared concentration were listed as Awarn.@ Reported data that were <80% or >120% of the prepared concentration were listed as Afail.@

When comparing reported concentrations to those prepared, the lower concentration ranges had more data that fell in Awarn@ and Afail@ categories than the higher level concentrations, i.e., there was less accuracy at the lower concentration ranges (Table 3). The acceptance criteria

for low concentration samples are quite narrow. For example, the summer 2006 blind audit of 0.00893 mg P/L prepared for orthophosphate has a Apass@ category (∀10%) of only 0.0080 - 0.0098 mg P/L. Eight out of eleven participating laboratories reported results that fell in the Awarn@ and Afail@ categories, indicating that their reported concentrations were greater than ∀10% of the prepared concentration in this low range. These results could be interpreted as an inability for most participants to accurately measure low level orthophosphate from concentrates provided to them. It would be important to know if there is also a difficulty in measuring natural low level samples. An alternative interpretation would be that it may be appropriate to broaden the acceptance boundaries for very low concentrations of prepared samples. There was also a broad range in low level orthophosphate summer 2006 reported data based on comparisons with other participants (mean 0.0098, S.D. 0.0018, C.V. 18.5%).

As with all past blind audits, the standard deviations for the low level ammonium samples were less than those for the higher level ammonium samples. However, the proportions of the standard deviations to the means for the low level ammonium samples were, again, quite large; i.e., coefficients of variation were 16% for 0.042 mg NH4-N/L (Summer 2006) and 39% for 0.036 mg NH4-N/L (Winter 2007). The coefficient of variation for the .026 mg N/L ammonium sample for winter 2002 was 20%. The coefficient of variation for the .0273 mg N/L ammonium sample for winter 2003 was 15%. As we reported in past years, the large variation in reported concentrations of low level ammonium for these blind audits probably indicates that inter-laboratory comparisons of any ammonium data prepared by laboratories from concentrates below 0.031 mg N/L would still be unreliable. These data indicate that comparison of multi-laboratory natural sample data below 0.042 mg NH4-N/L might also be unreliable.

There were thirteen instances where concentrations reported for dissolved constituents fell in the Awarn@ or Afail@ category based on the standard deviation of all participants= reported concentrations and also in the Awarn@ or Afail@ category based on percent recovery. These are listed for the individual laboratories in Appendix 1.

Acceptance Limits of Provided Particulate Samples: For each study, particulate samples were filtered from a common estuarine water sample and, consequently, are not true blind audit samples made from pure constituents. There is no Atrue@ or prepared concentration with which to compare. Except for the particulate nitrogen data for the winter 2007 audit, the standard deviation was less than 10% of the mean reported concentration for particulate carbon and nitrogen.

Data for particulate carbon and nitrogen at one laboratory for the blind audits in summer 2005 and winter 2006 were not in close agreement with the other participants= data. In summer 2004, their results were in agreement with other participants. They had no particulate carbon and nitrogen data for winter 2005. For summer 2006, their results were in agreement with other participants. However, for winter 2007, their particulate carbon data were in agreement with the other participants, but their particulate nitrogen data were about double those of the other participants.

Over the years, the concentration of particulate constituents provided to the participants has varied randomly over approximately a five-fold range. For example, particulate carbon in winter 1998 was approximately 0.45 mg C/L, and in summer 2002 was approximately 2.34 mg C/L. The proportions of the standard deviations to the means for particulate phosphorus were low (6.1%) for the summer 2006 blind audit, and for the winter 2007 blind audit (8.7%). The proportion of the standard deviation to the mean had been high for particulate phosphorus in both 2002 blind audits. This contrasted to most previous years of blind audits in which the

coefficient of variation for particulate phosphorus was the lowest of the particulate fractions. In both 2002 blind audits, one or two laboratories= reported concentrations were visibly different from the mean, thus increasing the coefficient of variation. The sample sizes were only five or seven, so it was not surprising that these differences were insufficient to generate a warning. These particulate phosphorus data comparisons are an obvious example of the danger of circular reasoning in these statistical assessments. The data being evaluated are also the data that were used to calculate the mean and standard deviation to which the data are being compared. New participants had been added to the blind audit program in 2001 and 2002; however, no laboratory expressed uncertainty in its reported particulate phosphorus concentrations. No laboratory reported concentrations for particulate phosphorus that were consistently different from the range of the other reported concentrations for both 2002 blind audits. All participants= reported concentrations were quite similar for the winter 2003 through winter 2007 blind audits, leading us to conclude that inter-laboratory comparison of other particulate phosphorus data would be valid.

Reporting Data Accurately: The data originally reported by all participants for the summer 2006 blind audit appeared, on casual inspection, to be reported accurately. In fact, no participant noted any discrepancies when all were contacted to review their data. A few of the results for the winter 2007 blind audit were miscalculated (and later corrected), or had Aslipped a decimal@ or exhibited some other obvious entry error that could have been easily avoided. As in past years, contacting the participants usually resolved these reporting discrepancies, but has not always improved their subsequent reporting practices. Other subtle entry or calculation errors may have gone undetected.

The number of significant figures reported in analytical results can significantly affect data comparability in a blind audit study. If a laboratory reports only two significant figures (for whatever reasons) and an audit sample has a prepared concentration expressed in three significant figures, then substantial under or over estimates of the comparative concentration can be reported. For example, if a 0.032 mg P/L sample has been prepared and a laboratory only reports two significant figures, i.e., 0.03 mg P/L, then the results expressed are 86% of the prepared value. During the 2000 study, all participants reported three significant digits for most parameters. It is noteworthy that the 2000 study's coefficients of variation were, generally, smaller than in the previous two years, probably a result of comparisons of data containing the appropriate number of significant digits. Unfortunately, some 2001, 2002, 2003, 2005, 2006 and winter 2007 participants reported only two significant digits for some analytes, thus potentially giving substantial under or over estimates for the comparisons.

CONCLUSION

Now that nineteen rounds of the Blind Audit Program have been completed, some consistent patterns have been observed that warrant action or further investigation:

1. Reported concentrations of analytes were usually similar between laboratories participating in the Blind Audit Program. No laboratory reported concentrations for individual analytes that were widely different from the range of the other reported concentrations for both blind audits. This indicates that most participating laboratories execute and report these measurements with accuracy and precision, reporting the appropriate number of significant digits.

2. When comparing reported concentrations to those prepared, the lower concentration ranges had more data that fell beyond ∀10% of the prepared sample than the higher level concentration ranges, i.e., there was less accuracy at the lower concentration ranges. This was particularly apparent for ammonium. The categories for Apass, warn and fail@ for low

concentration samples are quite narrow. Therefore, for very low concentrations of prepared samples, it may be appropriate to broaden the acceptance boundaries.

3. The large variation in reported concentrations of low level ammonium for both blind audits and several previous audits, probably indicates that inter-laboratory comparisons of any ammonium data prepared from concentrates below 0.042 mg N/L would be unreliable. It would be important to know if there is also a difficulty in measuring natural low level samples.

4. For all but two of the participating laboratories, there was remarkable consistency in the measurement of total suspended solids from the suspensions of infusorial earth; however, there was a consistent negative bias in the measurements, when compared to the prepared concentrations. This occurred in past years as well. Further checks will be made of the preparation steps for subsampling the suspensions that are sent to participants.

5. The proportion of the standard deviation to the mean was small for particulate phosphorus for the winter 2003 through winter 2007 blind audits, so inter-laboratory comparison of other particulate phosphorus data should be valid. The proportion of the standard deviation to the mean had been high for particulate phosphorus in both blind audits in 2001 and 2002. This contrasted to all three previous years, in which the coefficient of variation for particulate phosphorus was usually the lowest of the particulate fractions.

6. Care should continue to be taken when completing report forms. For the summer 2006 and winter 2007 blind audits, some results were AGAIN (!) reported with insufficient significant digits. For the summer 2006 blind audit, for the first time in at least 5 years, no results were reported and then later corrected. However, for the winter 2007 blind audits, some results were miscalculated (and later corrected). Over the course of the years, a few laboratories have repeatedly made calculation errors that were later corrected. It is hoped that the corrections of these lapses will serve as reminders of the importance to continuously check many aspects of data management to ensure overall data quality.

Table 1. Participants in the Summer 2006 and Winter 2007 Blind Audit Program.

Institution	Contact Person	Phone	Dissolved	Particulate	Chlorophyll a	DOC	TSS
Old Dominion University, Water Quality Lab, (**ODU**)	Suzanne Doughton	757-451-3043	X	X	X		X
University of MD, Horn Point Laboratory (**HPL**)	Lois Lane	410-221-8252	X	X		X	
Virginia Institute of Marine Science (**VIMS**)	Carol Pollard	804-684-9749	X	X	X		X
Virginia Div, Consolidated Lab Services (**DCLS**)	Jay Armstrong	804-648-4480 x328	X	X	X	X	X
Virginia Tech. Occoquan Lab (**OCC**)	Mary Lou Daniel	703-361-5606	X	X	X	X	X
MD Dept Health and Mental Hygiene (**DHMH**)	Asoka Katumuluwa	410-767-5034	X	X	X	X	X
Univ. of MD Chesapeake Bio Lab (**CBL**)	Carl Zimmermann	410-326-7252	X	X	X	X	X
University of Delaware (**UDEL**)	Joe Scudlark	302-645-4300	X	X			X
Delaware Dept. of Natural Resources (**DELDNR**)	Ben Pressly	302-739-4771	X		X	X	X
Morgan State University. Estuarine Research Center (**ANSERC**)	Richard Lacouture	410-586-9700			X		
Academy of Natural Science of Philadelphia (**PAACAD**)	Paul Kiry	215-299-1076	X	X	X	X	X
USGS National Water Quality Lab (**USGS**)	Mary Cast (sent no data)	303-236-3463	X	X	X	X	
PA DEP, Bureau of Laboratories (**PADEP**)	James Yoder	717-795-2425	X				X
MWRA, Water Quality Laboratory (**MWRA**)	Jennifer Prasse	617-860-7808	X	X	X	X	X
Hampton Roads Sanitation District (**HRSD**)	Stacie Metzler	757-460-4217	X			X	X

Table 2. Summary of Mean Concentration and Standard Deviation for Each Group of Analytes in the Summer 2006 and the Winter 2007 Blind Audit, Including Distribution of Reported Concentrations from the Mean.

Parameter	Concentration in mg/L		**Number of Laboratories**			
			Standard Deviations from Mean			
			<1	1-2	2-3	>3
	Mean	S.D.	**PASS**	**PASS**	**WARN**	**FAIL**
Summer 2006						
Total Dissolved Nitrogen	0.350	0.0346	8	2		
Total Dissolved Nitrogen	0.664	0.0621	8	2	1	
Total Dissolved Phosphorus	0.0317	0.0042	9		1	
Total Dissolved Phosphorus	0.0501	0.0062	9	2		
Ammonium	0.0397	0.0063	7	4		
Ammonium	0.340	0.0119	9	3		
Nitrate + Nitrite	0.0527	0.0067	8	1	1	
Nitrate + Nitrite	0.717	0.0207	9	2	1	
Orthophosphate	0.0098	0.0018	7	4		
Orthophosphate	0.0372	0.0017	8	4		
Dissolved Organic Carbon	1.75	0.314	8		1	
Dissolved Organic Carbon	4.82	0.180	7	1	1	
Particulate Carbon	1.64	0.0534	7	3		
Particulate Nitrogen	0.264	0.0123	6	4		
Particulate Phosphorus	0.0232	0.0014	5	2		
Total Suspended Solids	13.7	1.28	8	3		
Winter 2007						
Total Dissolved Nitrogen	0.333	0.035	4	5		1
Total Dissolved Nitrogen	0.6744	0.0571	8	3	1	
Total Dissolved Phosphorus	0.0164	0.0013	5	4	1	
Total Dissolved Phosphorus	0.0552	0.0186	11		1	
Ammonium	0.029	0.0114	9	1		
Ammonium	0.129	0.0162	9	4		
Nitrate + Nitrite	0.031	0.0028	9	1	1	
Nitrate + Nitrite	0.138	0.0075	8	4	1	
Orthophosphate	0.0050	0.0013	8	2	1	
Orthophosphate	0.0180	0.0017	9	3	1	
Dissolved Organic Carbon	2.28	0.1605	9		1	
Dissolved Organic Carbon	3.64	0.170	7	2	1	
Particulate Carbon	1.18	0.094	8	1	1	
Particulate Nitrogen	0.215	0.0596	9		1	
Particulate Phosphorus	0.0166	0.0014	6	3		
Total Suspended Solids	11.08	2.03	9	2	1	

Table 3. Summary of Prepared and Reported Concentrations for Each Analyte, Including Percent Recovery of the Prepared Concentration

			Number of Laboratories		
Parameter	Prepared Concentration mg/L	Reported Concentration Range mg/L	Within 90% - 110% of Prepared Concentration	Within 80 -90%, or 110-120% of Prepared Concentration	<80%, or >120% of Prepared Concentration
			PASS	**WARN**	**FAIL**
Summer 2006					
Total Dissolved Nitrogen	0.355	0.283-0.392	7	2	1
Total Dissolved Nitrogen	0.667	0.536-.748	8	3	
Total Dissolved Phosphorus	0.0288	0.0277-0.0414	6	3	1
Total Dissolved Phosphorus	0.0461	0.04-0.0621	5	4	2
Ammonium	0.0420**	0.030-0.0489	4	5	2
Ammonium	0.357	0.316-0.362	11	1	
Nitrate + Nitrite	0.0490	0.0461-0.0672	7	1	2
Nitrate + Nitrite	0.728	0.687-0.762	12		
Orthophosphate	0.0089**	0.0074-0.0129	3	4	4
Orthophosphate	0.0410	0.0339-0.039	7	5	
Dissolved Organic Carbon	1.50	1.55-2.56	4	3	2
Dissolved Organic Carbon	4.60	4.59-5.19	8	1	
Total Suspended Solids	15.0	12.0-16.3	6	5	1
Winter 2007					
Total Dissolved Nitrogen	0.310	0.198-0.404	6	1	3
Total Dissolved Nitrogen	0.639	0.6-0.96	10	1	1
Total Dissolved Phosphorus	0.0154**	0.0125-0.0234	5	4	1
Total Dissolved Phosphorus	0.0461	0.04-0.0564	8	2	2
Ammonium	0.036**	0.0259-0.075	8	1	1
Ammonium	0.139	0.29-0.369	7	4	2
Nitrate + Nitrite	0.0310	0.0409-0.055	9	1	1
Nitrate + Nitrite	0.140	0.7922-0.848	12	1	
Orthophosphate	0.0045**	0.0082-0.013	1	4	6
Orthophosphate	0.0164	0.0165-0.022	7	5	1
Dissolved Organic Carbon	2.20	1.60-2.3	8	2	
Dissolved Organic Carbon	3.60	3.7-4.21	10		
Total Suspended Solids	13.0	3.95-10	6	3	3

**For very low concentrations of prepared samples, it may be appropriate to broaden the acceptance boundaries.

Appendix 1. Summer 2006 and Winter 2007 Reported Data, Prepared Concentrations and Percent Recoveries. Warnings based on standard deviation of the mean of reported concentrations are listed.

Virginia Institute of Marine Science

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.378	.355	106.5	.210 FAIL	.310	67.7
TDN (mg N/L)	.693	.667	103.9	.5845	.639	91.5
TDP (mg P/L)	.0301	.0288	104.5	.0203 WARN	.0154	131.8**
TDP (mg P/L)	.0561	.0461	121.7	.0477	.0461	103.5
NH4 (mg N/L)	.0444	.042	105.7**	.0157	.036	43.6**
NH4 (mg N/L)	.0345	.357	96.6	.121	.139	87.1
NO3 + NO2 (mg N/L)	.0525	.0490	107.1	.0336	.0310	108.4
NO3 + NO2 (mg N/L)	.735	.728	101.0	.119 WARN	.140	85.0
PO4 (mg P/L)	.0109	.0089	122.5**	.0035	.0045	77.8**
PO4 (mg P/L)	.0386	.0410	94.1	.0174	.0164	106.1
Particulate C (mg C/L)	1.72			1.17		
Particulate N (mg N/L)	.270			.182		
Particulate P (mg P/L)				.0143		
Chlorophyll (μg/L)	5.26			.42		
Total Suspended Solids (mg/L)	12.0	15.0	80.0	12.9	13.0	99.2

"WARN@ and AFAIL@ based on standard deviation of all participants= reported concentrations

**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.

Appendix I. *Continued.*

Occoquan Watershed Monitoring Laboratory

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.345	.355	97.2	.376	.310	121.3
TDN (mg N/L)	.681	.667	102.1	.700	.639	109.5
TDP (mg P/L)	.034	.0288	118.1	.018	.0154	116.9**
TDP (mg P/L)	.052	.0461	112.8	.048	.0461	104.1
NH4 (mg N/L)	.048	.042	114.3**	.036	.036	100.0**
NH4 (mg N/L)	.332	.357	93.0	.135	.139	97.1
NO3 + NO2 (mg N/L)	.055	.0490	112.2	.024 WARN	.0310	77.4
NO3 + NO2 (mg N/L)	.700	.728	96.2	.138	.140	98.6
PO4 (mg P/L)	.012	.0089	134.8**	.004	.0045	88.9**
PO4 (mg P/L)	.038	.0410	92.7	.014 WARN	.0164	85.4
Particulate C (mg C/L)	1.61			1.40 WARN		
Particulate N (mg N/L)	.244			.380 WARN		
Chlorophyll (μg/L)	13.5			5.2		
DOC (mg C/L)	1.61	1.50	107.3	1.91 WARN	2.20	86.8
DOC (mg C/L)	4.79	4.60	104.1	3.32	3.60	92.2
Total Suspended Solids (mg/L)	16.3	15.0	108.7	12.4	13.0	95.4

AWARN@ based on standard deviation of all participants= reported concentrations
**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.

Appendix I. *Continued.*

Delaware DNR

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.283	.355	79.7	.381	.310	122.9
TDN (mg N/L)	.536 WARN	.667	80.4	.650	.639	101.7
TDP (mg P/L)	.0277	.0288	96.2	.0170	.0154	110.4**
TDP (mg P/L)	.0458	.0461	99.3	.0481	.0461	104.3
NH4 (mg N/L)	.0489	.042	116.4**	Not determined	.036	
NH4 (mg N/L)	.362	.357	101.4	.161	.139	115.8
NO3 + NO2 (mg N/L)	.0608	.0490	124.1	.0344	.0310	111.0
NO3 + NO2 (mg N/L)	.742	.728	101.9	.138	.140	98.6
PO4 (mg P/L)	.0113	.0089	127.0**	.00792 WARN	.0045	176.0**
PO4 (mg P/L)	.0364	.0410	88.8	.0211	.0164	128.7
Particulate C (mg C/L)	1.61			1.18		
Particulate N (mg N/L)	.254			.192		
Particulate P (mg P/L)	.0239			.01555		
Chlorophyll (μg/L)	13.9			8.30		
DOC (mg C/L)	1.58	1.50	105.3	2.36	2.20	107.3
DOC (mg C/L)	4.59	4.60	99.8	3.50	3.60	97.2
Total Suspended Solids (mg/L)	15.0	15.0	100.0	11.6	13.0	89.2

AWARN@ based on standard deviation of all participants= reported concentrations
**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.

Appendix I. *Continued.*

University of Delaware

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
NH4 (mg N/L)	.0366	.042	87.1**	.0384	.036	106.7**
NH4 (mg N/L)	.341	.357	95.5	.137	.139	98.6
NO3 + NO2 (mg N/L)	.0461	.0490	94.1	.0330	.0310	106.5
NO3 + NO2 (mg N/L)	.724	.728	99.5	.147	.140	105.0
PO4 (mg P/L)	.0078	.0089	87.6**	.0054	.0045	120.0**
PO4 (mg P/L)	.0367	.0410	89.5	.0187	.0164	114.0

**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.

Appendix I. *Continued.*

Academy of Natural Sciences of Philadelphia

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.317	.355	89.3	.298	.310	96.1
TDN (mg N/L)	.614	.667	92.1	.601	.639	94.1
TDP (mg P/L)	.0293	.0288	101.7	.0142	.0154	92.2**
TDP (mg P/L)	.0471	.0461	102.2	.0443	.0461	96.1
NH4 (mg N/L)	.0399	.042	95.0**	.0328	.036	91.1**
NH4 (mg N/L)	.330	.357	92.4	.128	.139	92.1
NO3 + NO2 (mg N/L)	.0498	.0490	101.6	.0310	.0310	100.0
NO3 + NO2 (mg N/L)	.697	.728	95.7	.135	.140	96.4
PO4 (mg P/L)	.0082	.0089	92.1**	.0054	.0045	120.0**
PO4 (mg P/L)	.0352	.0410	85.9	.0179	.0164	109.1
Particulate C (mg C/L)	1.66			1.18		
Particulate N (mg N/L)	.249			.1865		
Particulate P (mg P/L)	.0257			.0184		
Chlorophyll (μg/L)	17.1			5.28		
DOC (mg C/L)	1.69	1.50	112.7	2.12	2.20	96.4
DOC (mg C/L)	4.93	4.60	107.2	3.55	3.60	98.6
Total Suspended Solids (mg/L)	14.2	15.0	94.7	10.9	13.0	83.8

**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.

Morgan State University Estuarine Research Center

Parameter	Summer 2006 Reported	Prepared	% Recovered	Winter 2007 Reported	Prepared	% Recovered
Chlorophyll (μg/L)	9.6			4.27		

Appendix I. *Continued.*

Old Dominion University

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.392	.355	110.4	.335	.310	108.1
TDN (mg N/L)	.722	.667	108.2	.665	.639	104.1
TDP (mg P/L)	.0344	.0288	119.4	.0158	.0154	102.6**
TDP (mg P/L)	.0550	.0461	119.3	.0501	.0461	108.7
NH4 (mg N/L)	.0399	.042	95.0**	.0349	.036	96.9**
NH4 (mg N/L)	.3478	.357	97.4	.1498	.139	107.8
NO3 + NO2 (mg N/L)	.0493	.0490	100.6	.0313	.0310	101.0
NO3 + NO2 (mg N/L)	.7257	.728	99.7	.1402	.140	100.1
PO4 (mg P/L)	.0102	.0089	114.6**	.0067	.0045	148.9**
PO4 (mg P/L)	.0386	.0410	94.1	.0157	.0164	95.7
Particulate C (mg C/L)	1.74			1.138		
Particulate N (mg N/L)	.260			.1978		
Particulate P (mg P/L)	.0226			.0171		
Chlorophyll (μg/L)	13.5			7.2		
Total Suspended Solids (mg/L)	14.3	15.0	95.3	13.34	13.0	102.6

**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.

Appendix I. *Continued.*

Virginia Division of Consolidated Laboratory Services

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.364	.355	102.5	.297	.310	95.8
TDN (mg N/L)	.735	.667	110.2	.605	.639	94.7
TDP (mg P/L)	.031	.0288	107.6	.016	.0154	103.9**
TDP (mg P/L)	.050	.0461	108.5	.049	.0461	106.3
NH4 (mg N/L)	.030	.042	71.4**	.034	.036	94.4**
NH4 (mg N/L)	.330	.357	92.4	.119	.139	85.6
NO3 + NO2 (mg N/L)	.048	.0490	98.0	.030	.0310	96.8
NO3 + NO2 (mg N/L)	.710	.728	97.5	.146	.140	104.3
PO4 (mg P/L)	.010	.0089	112.4**	.006	.0045	133.3**
PO4 (mg P/L)	.039	.0410	95.1	.019	.0164	115.9
Particulate C (mg C/L)	1.59			1.09		
Particulate N (mg N/L)	.266			.187		
Particulate P (mg P/L)	.0228			.0169		
Chlorophyll (μg/L)	11.0			6.57		
DOC (mg C/L)	2.56 WARN	1.50	170.7	2.44	2.20	110.9
DOC (mg C/L)	4.78	4.60	103.9	3.29 WARN	3.60	91.4
Total Suspended Solids (mg/L)	13	15.0	86.7	13	13.0	100.0

**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.
AWARN@ based on standard deviation of all participants= reported concentrations

Hampton Roads Sanitation District

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.65	.667	97.5	.70	.639	109.5
TDP (mg P/L)	.04	.0461	86.8	.11 WARN	.0461	238.6
NH4 (mg N/L)	.35	.357	98.0	.11	.139	79.1
NO3 + NO2 (mg N/L)	.71	.728	97.5	.14	.140	100.0
PO4 (mg P/L)	.039	.0410	95.1	.018	.0164	109.8
DOC (mg C/L)	1.67	1.50	111.3	2.26	2.20	102.7
DOC (mg C/L)	5.19 WARN	4.60	112.8	3.69	3.60	102.5
Total Suspended Solids (mg/L)	14	15.0	93.3	11	13.0	84.6

AWARN@ based on standard deviation of all participants= reported concentrations.

PADEP Water Quality Laboratory

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)				0.80 WARN	.639	125.2
TDP (mg P/L)				0.053	.0461	115.0
NH4 (mg N/L)				0.1	.139	71.9
NO3 + NO2 (mg N/L)				0.14	.140	100.0
PO4 (mg P/L)				0.018	.0164	109.8
DOC (mg C/L)				2.40	2.20	109.1
DOC (mg C/L)				3.65	3.60	101.4
Total Suspended Solids (mg/L)				7 WARN	13.0	53.8

AWARN@ based on standard deviation of all other participants= reported concentrations.

Appendix I. *Continued.*

UMCES Horn Point Laboratory

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.377	.355	106.2	.372	.310	120.0
TDN (mg N/L)	.748	.667	112.1	.720	.639	112.7
TDP (mg P/L)	.0321	.0288	111.5	.0169	.0154	109.7**
TDP (mg P/L)	.0515	.0461	111.7	.0539	.0461	116.9
NH4 (mg N/L)	.0318	.042	75.7**	.0336	.036	93.3**
NH4 (mg N/L)	.3163	.357	88.6	.136	.139	97.8
NO3 + NO2 (mg N/L)		.0490		.0315	.0310	101.6
NO3 + NO2 (mg N/L)	.723	.728	99.3	.140	.140	100.0
PO4 (mg P/L)	.0074	.0089	83.1**	.0040	.0045	88.9**
PO4 (mg P/L)	.0366	.0410	89.3	.0177	.0164	107.9
Particulate C (mg C/L)	1.64			1.11		
Particulate N (mg N/L)	.280			.194		
Particulate P (mg P/L)				.0189		
Chlorophyll (μg/L)	15.2			6.42		
DOC (mg C/L)	1.55	1.50	103.3	2.24	2.20	101.8
DOC (mg C/L)	4.68	4.60	101.7	3.68	3.60	102.2
Total Suspended Solids (mg/L)	12.0	15.0	80.0	8.04	13.0	61.8

**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.

Appendix I. *Continued.*

UMCES Chesapeake Biological Laboratory

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.337	.355	94.9	.318	.310	102.6
TDN (mg N/L)	.642	.667	96.3	.661	.639	103.4
TDP (mg P/L)	.0288	.0288	100.0	.0149	.0154	96.8**
TDP (mg P/L)	.0477	.0461	103.5	.0476	.0461	103.3
NH4 (mg N/L)	.045	.042	107.1**	.034	.036	94.4**
NH4 (mg N/L)	.360	.357	100.8	.118	.139	84.9
NO3 + NO2 (mg N/L)	.0510	.0490	104.1	.0327	.0310	105.5
NO3 + NO2 (mg N/L)	.762 WARN	.728	104.7	.146	.140	104.3
PO4 (mg P/L)	.0129	.0089	144.9**	.0043	.0045	95.6**
PO4 (mg P/L)	.0339	.0410	82.7	.0157	.0164	95.7
Particulate C (mg C/L)	1.61			1.10		
Particulate N (mg N/L)	.278			.194		
Particulate P (mg P/L)	.0233			.0164		
Chlorophyll (µg/L)	12.8			6.46		
DOC (mg C/L)	1.82	1.50	121.3	2.41	2.20	109.5
DOC (mg C/L)	4.95	4.60	107.6	3.86	3.60	107.2
Total Suspended Solids (mg/L)	13.4	15.0	89.3	12.0	13.0	92.3

**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.
AWARN@ based on standard deviation of all participants= reported concentrations

Appendix I. *Continued.*

MD DHMH Division of Environmental Chemistry Nutrients Laboratory

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.325	.355	91.5	.297	.310	95.8
TDN (mg N/L)	.615	.667	92.2	.628	.639	98.3
TDP (mg P/L)	.0277	.0288	96.2	.0178	.0154	115.6**
TDP (mg P/L)	.0439	.0461	95.2	.0458	.0461	99.3
NH4 (mg N/L)	.0360	.042	85.7**	.0329	.036	91.4**
NH4 (mg N/L)	.344	.357	96.4	.129	.139	92.8
NO3 + NO2 (mg N/L)	.0672 WARN	.0490	137.1	.0306	.0310	98.7
NO3 + NO2 (mg N/L)	.709	.728	97.4	.137	.140	97.9
PO4 (mg P/L)	.00835	.0089	93.8**	.00597	.0045	132.7**
PO4 (mg P/L)	.0371	.0410	90.5	.0181	.0164	110.4
Particulate C (mg C/L)	1.59			1.276		
Particulate N (mg N/L)	.276			.230		
Particulate P (mg P/L)	.0213			.0155		
Chlorophyll (μg/L)	12.85			6.45		
DOC (mg C/L)	1.71	1.50	114.0	2.34	2.20	106.4
DOC (mg C/L)	4.81	4.60	104.6	3.79	3.60	105.3
Total Suspended Solids (mg/L)	13.55	15.0	90.3	12.05	13.0	92.7

**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.
AWARN@ based on standard deviation of all participants= reported concentrations.

Appendix I. *Continued.*

MWRA Water Quality Laboratory

Parameter	Summer 2006 Reported	Summer 2006 Prepared	% Recovered	Winter 2007 Reported	Winter 2007 Prepared	% Recovered
TDN (mg N/L)	.380	.355	107.0	.323	.310	104.2
TDN (mg N/L)	.668	.667	100.1	.688	.639	107.7
TDP (mg P/L)	.0414 WARN	.0288	143.8	.0173	.0154	112.3**
TDP (mg P/L)	.0621	.0461	134.7	.0579	.0461	125.6
NH4 (mg N/L)	.0367	.042	87.4**	.0321	.036	89.2**
NH4 (mg N/L)	.340	.357	95.2	.135	.139	97.1
NO3 + NO2 (mg N/L)	.0469	.0490	95.7	.0294	.0310	94.8
NO3 + NO2 (mg N/L)	.687	.728	94.4	.129	.140	92.1
PO4 (mg P/L)	.00909	.0089	102.1**	.00576	.0045	128.0**
PO4 (mg P/L)	.0363	.0410	88.5	.0181	.0164	110.4
Particulate C (mg C/L)	1.62			1.15		
Particulate N (mg N/L)	.262			.204		
Particulate P (mg P/L)	.0228			.0162		
Chlorophyll (μg/L)	14.8			8.44		
DOC (mg C/L)	1.58	1.50	105.3	2.30	2.20	104.5
DOC (mg C/L)	4.69	4.60	102.0	3.72	3.60	103.3
Total Suspended Solids (mg/L)	12.8	15.0	85.3	10.1	13.0	77.7

**The prepared sample concentration was quite low, so the acceptance boundaries are narrow.
AWARN@ based on standard deviation of all participants= reported concentration.

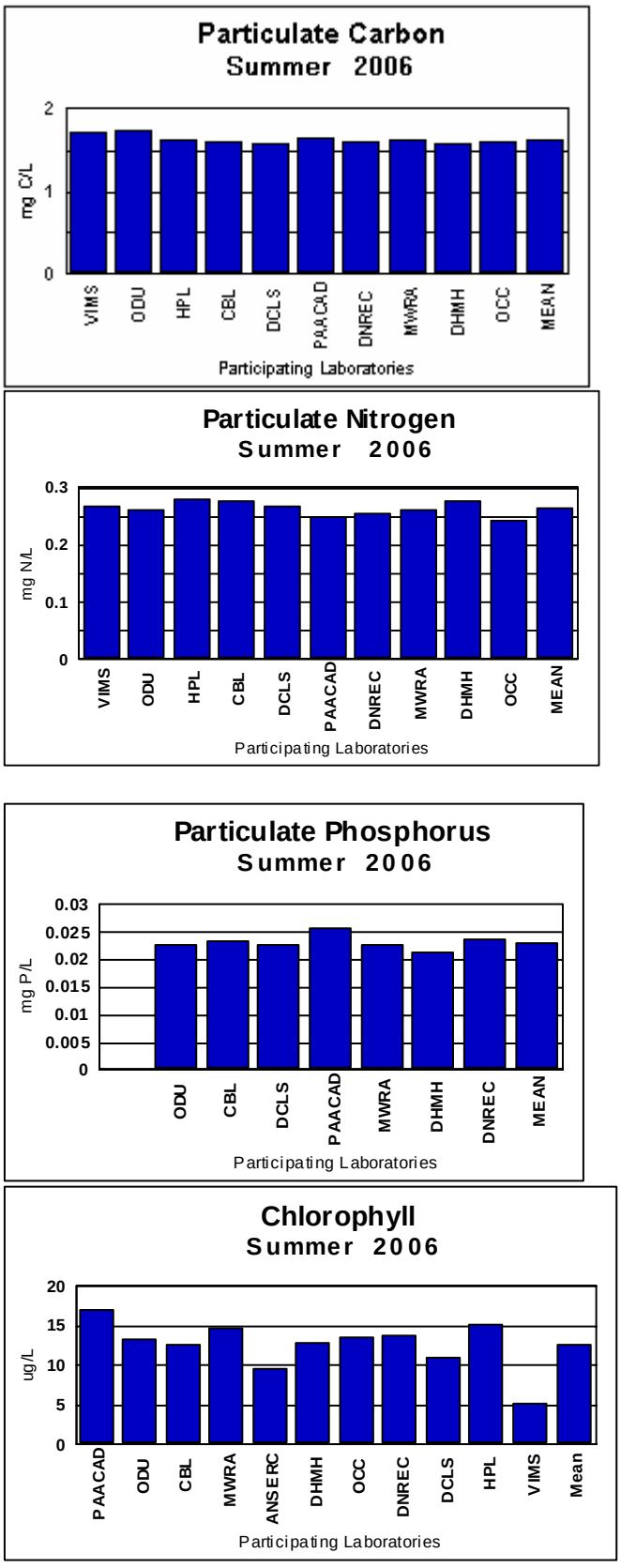


Figure 1. Particulate carbon, nitrogen and phosphorus; chlorophyll, Summer 2006.

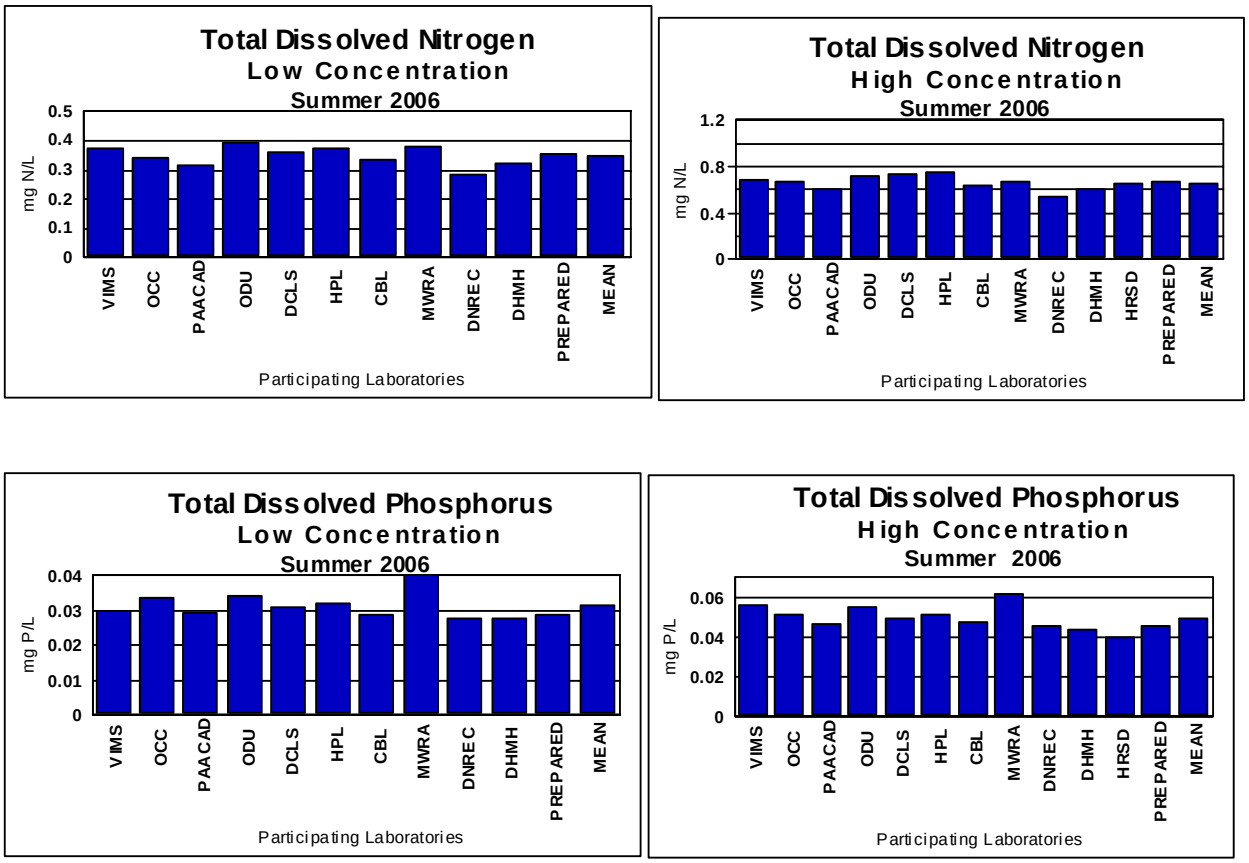
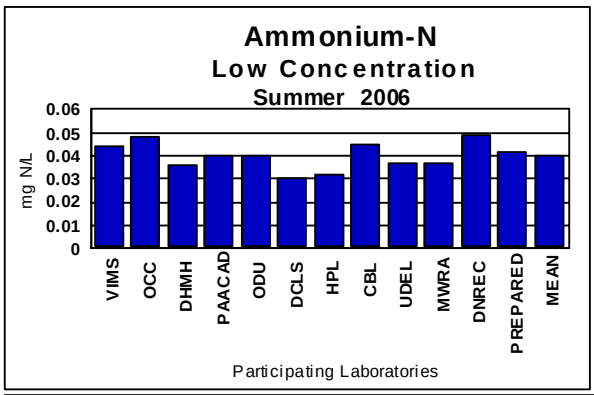
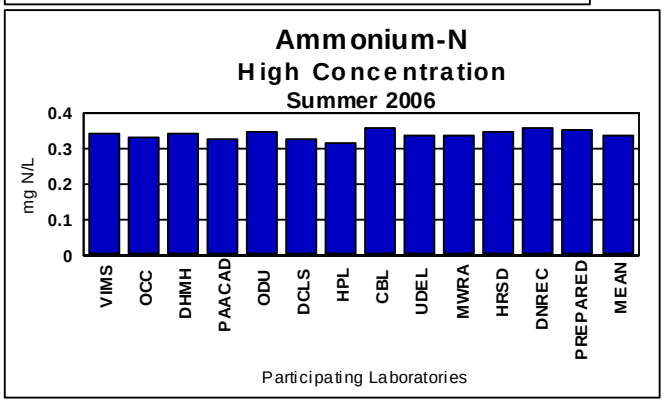
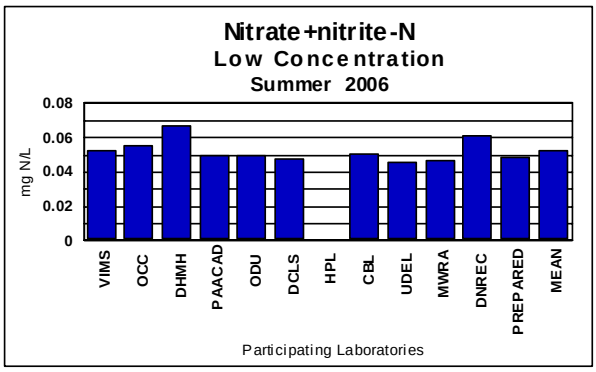


Figure 2. Total dissolved nitrogen and phosphorus, Summer 2006.







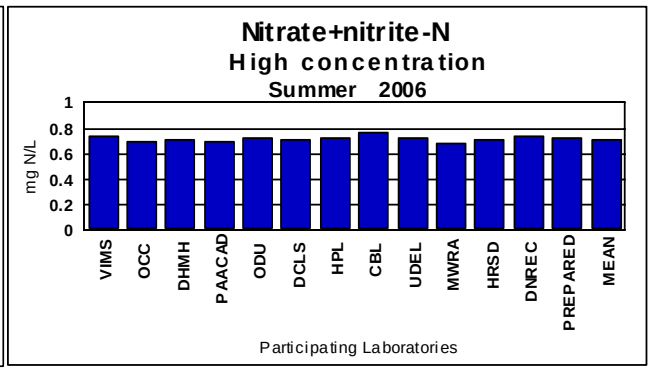


Figure 3. Dissolved inorganic nitrogen and phosphorus, Summer 2006.

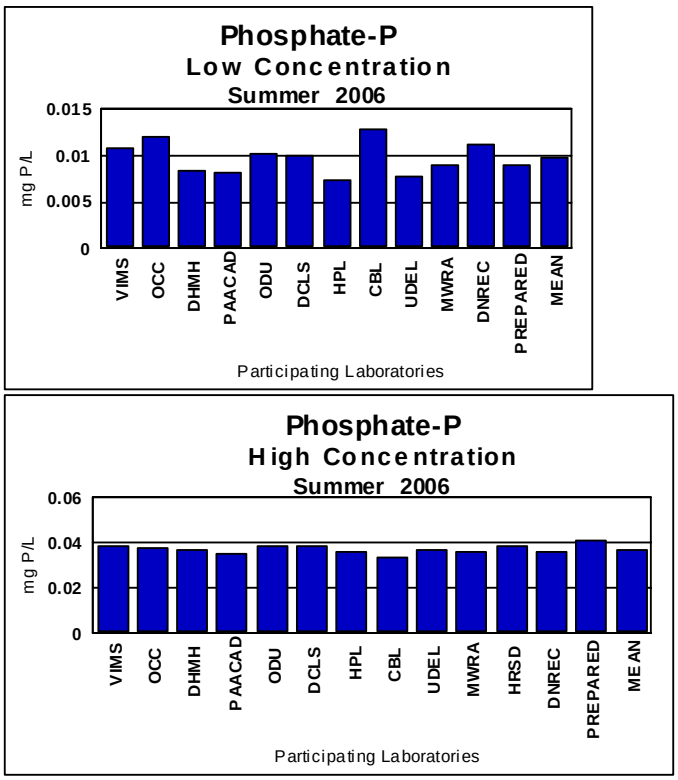
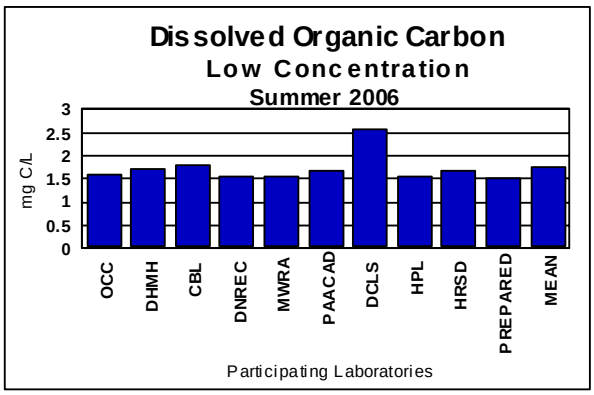
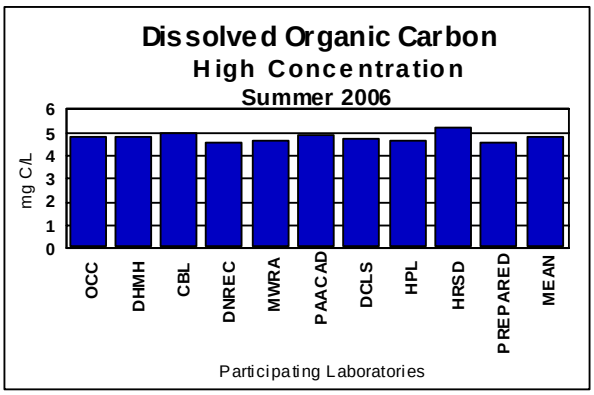


Figure 3 continued. Dissolved inorganic nitrogen and phosphorus, Summer 2006.





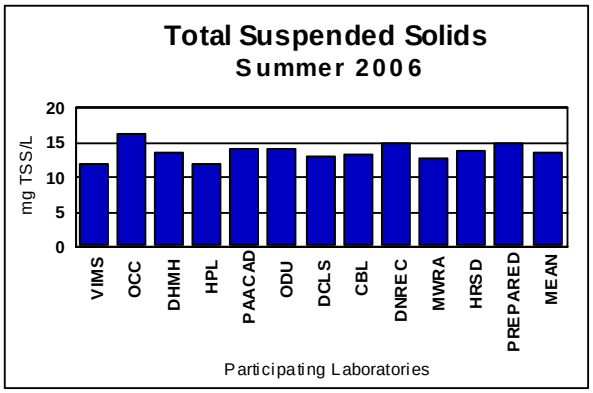


Figure 4. Dissolved organic carbon and total suspended solids, Summer 2006

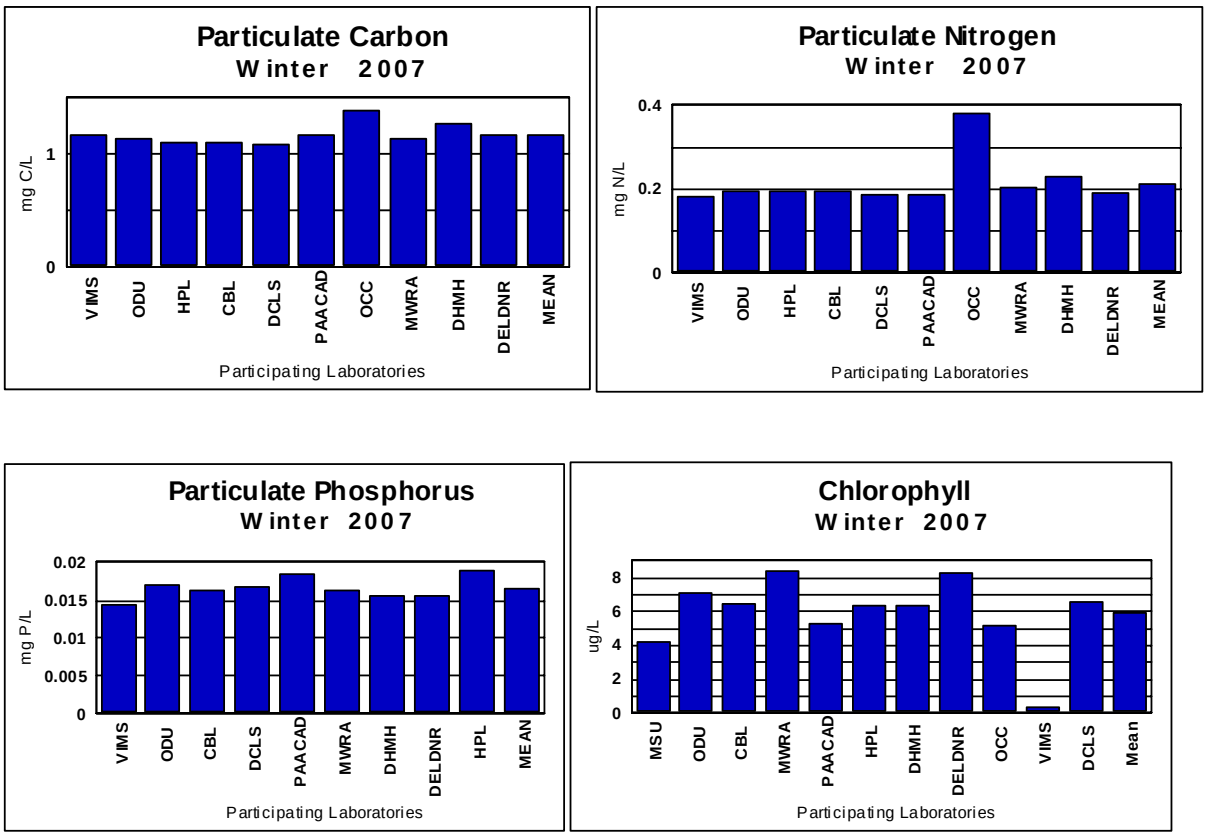


Figure 5. Particulate carbon, nitrogen and phosphorus; chlorophyll, Winter 2007.

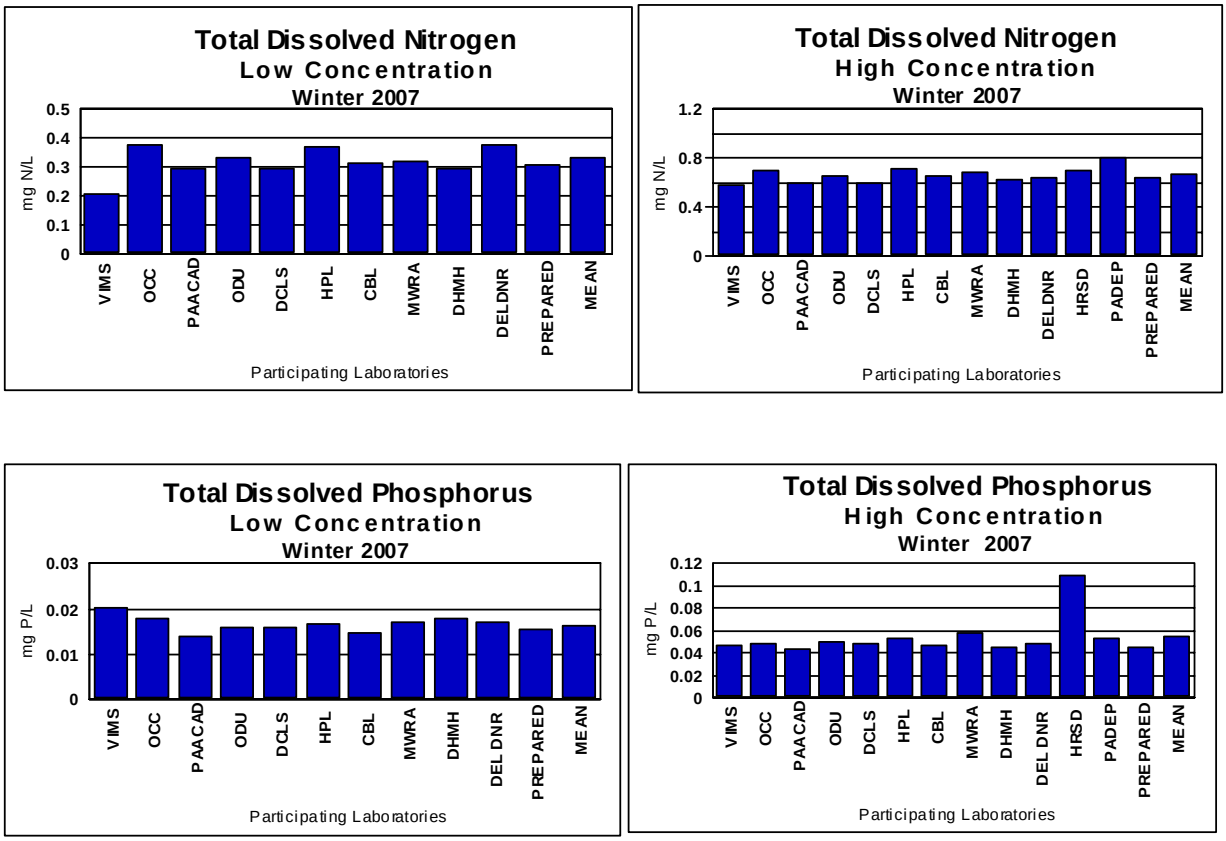


Figure 6. Total dissolved nitrogen and phosphorus, Winter 2007

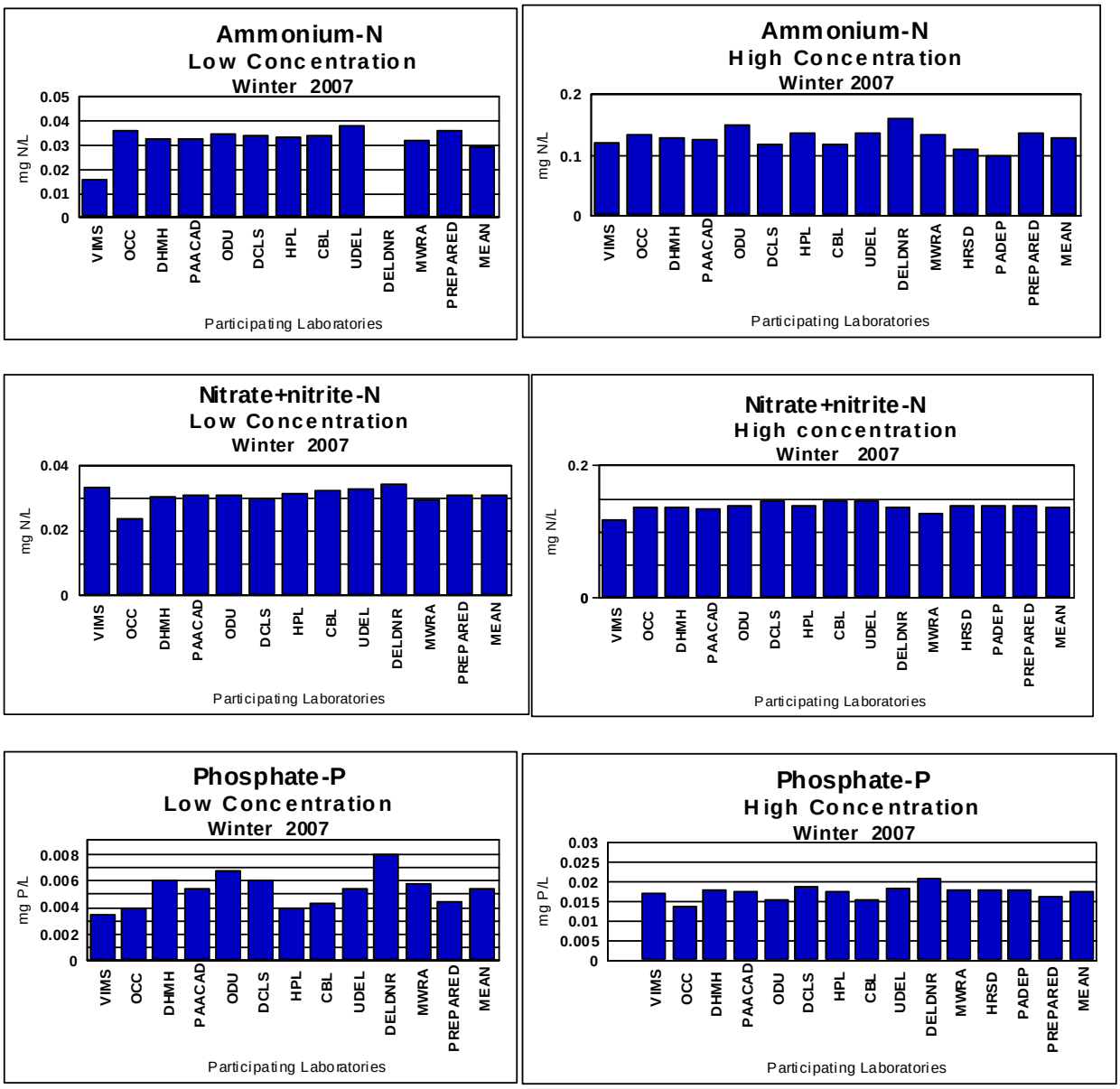
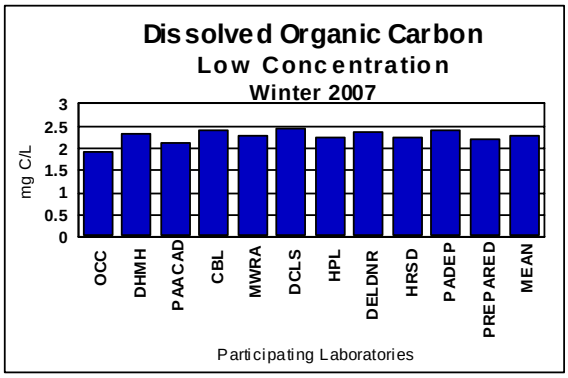
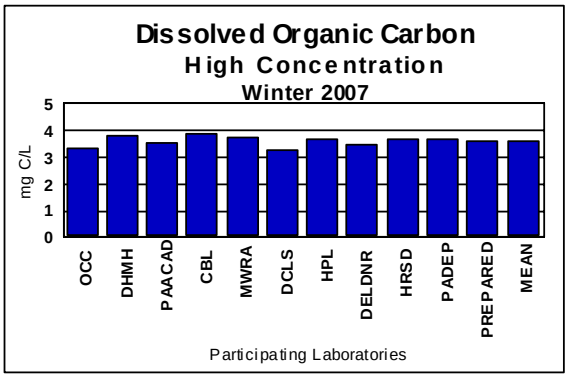


Figure 7. Dissolved inorganic nitrogen and phosphorus, Winter 2007.





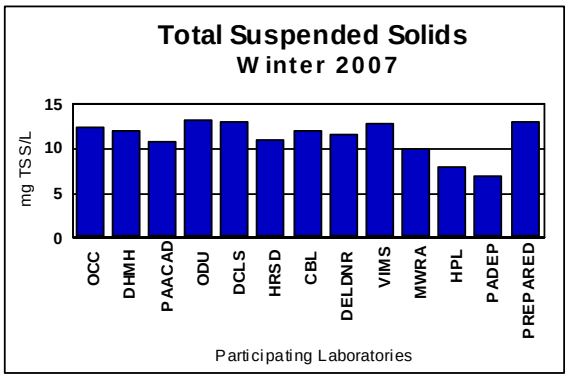


Figure 8. Dissolved organic carbon and total suspended solids, Winter 2007.