

FINAL REPORT

CHESAPEAKE BAY PROGRAM BLIND AUDIT

Fiscal Year 2016 Final Report

PREPARED FOR:

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Water and Habitat Quality Program
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October 2016

*Technical Report Series No. TS-687-16
of the University of Maryland Center for Environmental Science*

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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 National Pollutant Discharge Elimination System (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 eighteenth 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 30 September 2015 and 12 April 2016. Participating laboratories and contact personnel are found in Table 1.

Parameters measured were: total dissolved organic nitrogen, total dissolved organic phosphorus, nitrate+nitrite, ammonium, orthophosphate 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 *a* 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, 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 47°C) 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 a analysis were filtered from natural population samples onto 47 mm GF/F filter pads. Replicate pads were provided to participating laboratories.

Total suspended solids blind audits were prepared as follows: 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 2015 and winter 2016 audits are found at the end of the report. Shortly after the completion of the study, a brief data report was sent to each participant asking 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: Results from both the summer 2015 and winter 2016 audits were excellent. Low and high reported concentrations had mean values that closely reflected the prepared concentrations. For example, the reported low concentrations of total dissolved N for summer 2015 were extremely close to the prepared concentration (prepared low: 0.338 mg N/L with (remarkable) mean reported concentration of 0.339 mg N/L).

Total Dissolved Phosphorus: Most reported concentrations for both summer 2015 and winter 2016 samples were consistently close to other laboratories' reported concentrations and closely reflected the prepared concentrations; e.g., prepared low winter concentration of 0.0100 mg P/L with mean reported concentration of all participants of 0.0115 mg P/L.

Ammonium: Analysis of low level samples for summer 2015 provided a mean concentration of 0.026 mg N/L compared to the prepared concentration of 0.025 mg N/L. Low level winter 2016 results were similar with 0.019 mg N/L mean reported concentration compared to the prepared concentration of 0.017 mg N/L. Variation around that mean for low level ammonium reported concentrations resulted in coefficients of variation of 16% for summer 2015; 19% for winter 2016. Results for both summer 2015 and winter 2016 high level concentrations were in close agreement with prepared concentrations and other reported values. Coefficients of variation of less than 7% were obtained.

Nitrate + Nitrite: Particularly good agreement was found among most laboratories for low concentrations, resulting in a mean concentration of 0.0571 mg N/L for summer 2015 compared to the prepared concentration of 0.0561 mg N/L. Results for both summer 2015 and winter 2016 high level concentrations were in close agreement with other reported values. Mean concentrations closely approximated prepared concentrations and low standard deviations provided percent coefficients of variation of 3-8% for both low and high level concentrations.

Orthophosphate: Low level concentrations for summer 2015 and winter 2016 were somewhat variable, with coefficients of variation of 12% and 26%. Analysis of low level samples for summer 2015 provided a mean concentration of 0.0176 mg P/L compared to the prepared concentration of 0.0163 mg P/L. Low level winter 2015 results were similar with 0.0089 mg P/L mean reported concentration compared to the prepared concentration of 0.0074 mg P/L. Reported results of the high level concentrations were closer to the prepared concentration, with coefficients of variation of 3% and 4% (prepared high for winter 2015: 0.0928 mg P/L with mean reported concentration of 0.0932 mg P/L).

Dissolved Organic Carbon: Particularly good agreement was found among all laboratories for

low and high concentrations for audits. Coefficients of variation were 2-8% for both concentration ranges for all four audits.

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 4.

Particulate Carbon: Among laboratory agreement was close for both the summer 2015 and winter 2016 audits with coefficients of variation of only 5-7%.

Particulate Nitrogen: Results for particulate nitrogen followed the same pattern as particulate carbon for both audits with coefficients of variation 11-13%. One laboratory's reported concentrations were substantially higher than any another laboratory's reported concentration for both audits.

Particulate Phosphorus: Particulate phosphorus concentrations showed some variability between the participating laboratories with two laboratories reporting substantially different concentrations from the other participants. Coefficients of variation were 13% for the summer 2015 audit and 26% for winter 2016 audits.

Chlorophyll a: Most chlorophyll a results for the summer 2015 and winter 2016 audits displayed the usual close agreement that was remarkable for multi-laboratory comparison of low concentrations of an environmentally transitory compound. Results from one laboratory were about half those of the "consensus" concentrations for the winter 2016 audit. The coefficients of variation were 18% for the summer 2015 samples and 20% for the winter 2016 samples.

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 2015 sample, 44.7 mg/L was prepared and for the winter 2016 sample, 74.5 mg/L was prepared, obtaining coefficients of variation only 4% for each audit.

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 0.05 mg N/L. Any total dissolved nitrogen measurement has a potential 0.05 mg N/L variability associated with it. This variability, when expressed as a percent of the TRUE 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 25% associated with it; whereas, a 1.20 mg N/L concentration has an analytical variability of 4%.

Acceptance Limits of Provided Dissolved Samples: Companies that prepare large quantities of performance evaluation samples assign acceptable confidence limits around the TRUE 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 \pm two 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 (Environmental Resource Associates), 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 two standard deviations PASS and those greater than three standard deviations FAIL. Results between two and three standard deviations receive the WARN flag.

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 “reality” in small sample sets with little variability between individual values, that at least one value 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 “passed.” It should also be noted that approximately the same number were in the “warning” category as in most of the previous studies, and that only three values in the entire study “failed.”

Data sets with relatively small standard deviations yielded more potentially extraneous “warning” points. For example, in the summer 2015 blind audit of high level ammonium concentration, the prepared concentration was 0.361 mg N/L and the mean reported concentration was 0.365 mg N/L (!) and reported concentrations ranged from 0.337-0.395 mg N/L. The coefficient of variation was ONLY 4%! Thirteen laboratories reported results for this high level sample that were within two standard deviations (S.D. 0.014 mg N/L) of the mean. Since the standard deviation was so small, two laboratories’ reported results for this sample that were between two and three standard deviations of the mean, so were labeled WARN. Thus, by that measure of accuracy, most of the data “passed” and two were “warned.” This ammonium 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. All of the reported summer 2015 high level ammonium data were within 9% of the prepared concentration!

Data were also assessed by comparing reported concentrations to those that had been prepared (Table 3). Groupings of data in PASS, WARN, and FAIL categories were arbitrarily set. Reported data that were within 10% of the prepared concentration were considered as PASS. Reported data that were 80-90% or 110 -120% of the prepared concentration were tabulated as WARN. Reported data that were <80% or >120% of the prepared concentration were tabulated as FAIL.

When comparing reported concentrations to those prepared, the lower concentration ranges had more data that fell in WARN and FAIL 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, for winter 2016 blind audit of 0.014 mg

N/L prepared for ammonium has a PASS category ($\pm 10\%$) of only 0.013 - 0.015 mg N/L. For the winter 2016 blind audit, eight out of twelve participating laboratories reported results that fell in the WARN or FAIL category, indicating that their reported concentrations were greater than $\pm 10\%$ of the prepared concentration in this low range. These results could be interpreted as an inability for all participants to accurately measure low level ammonium from concentrates provided to them. It would be important to know if there is also 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 percentage recovery of low level ammonium reported values in past audits; however, when comparing with other participants, the coefficient of variation remains remarkably small. For example, winter 2016 reported data based on comparisons with other participants, the low level ammonium mean was 0.0189 mg N/L, S.D. 0.0035, C.V. 19%.

There was less divergence between participants for the summer 2012 through summer 2014 low level ammonium samples than in audits of summer 2011 and winter 2012. For these most recent prepared ammonium samples, the proportion of the standard deviation to the mean was approximately the same as it had been for the last few years. Variation around the mean for low level ammonium reported concentrations resulted in coefficients of variation of 16% for summer 2015 concentration of 0.025 mg NH₄-N/L; 19% for winter 2016 concentration of 0.014. For the winter 2014 audit, the coefficient of variation for 0.022 mg NH₄-N/L was 20% mg NH₄-N/L. The coefficient of variation was 16% for 0.042 mg NH₄-N/L (Summer 2006) and 39% for 0.036 mg NH₄-N/L (Winter 2007). This indicates that inter-laboratory comparisons of any ammonium data prepared by most laboratories from concentrates below 0.042 mg N/L could probably be somewhat improving!

There were twelve instances where concentrations reported for dissolved constituents or total suspended solids fell in the WARN or FAIL category based on the standard deviation of all participants' reported concentrations and also in the WARN or FAIL 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 carbon, nitrogen, phosphorus and chlorophyll *a* samples were filtered from a common estuarine water sample and, consequently, are not true blind audit samples made from pure constituents. There is no "true" or prepared concentration with which to compare. The standard deviation was less than 13% of the mean reported concentrations for particulate carbon and nitrogen for the summer 2015 and winter 2016 audits. One laboratory's reported particulate nitrogen concentration was nearly double that of all other laboratories' reported mean concentrations for the summer 2015 and winter 2016 audits, although their reported particulate carbon concentration was not substantially different than the mean of the other participants.

Particulate phosphorus concentrations showed some variability between the participating laboratories: coefficients of variation 13% for summer 2015 and 26% for winter 2016 audits. One laboratory's reported particulate phosphorus concentrations were substantially lower than all other laboratories' reported mean concentrations for both audits. Another laboratory's reported particulate phosphorus concentrations were substantially higher than all other laboratories' reported mean concentrations for both audits.

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 winter 2013 was approximately 2.35 mg C/L. Particulate phosphorus in winter 2014 was 0.0091 mg P/L and in winter 1999 was 0.0529 mg P/L.

Reporting Data Accurately: Most data originally reported by all participants for both these blind audits appeared, on casual inspection, to be reported accurately. Subtle entry or calculation errors may have gone undetected.

The summer 2007 and winter 2008 audits were the first pair of audits in which no participant noted any discrepancies when all were contacted to review their data. No results were miscalculated (and later corrected), or had “slipped a decimal” or exhibited some other obvious entry error that could have been easily avoided. After years of reporting “difficulties,” participants had improved their reporting practices! Sadly, this improvement in reporting did not extend to the summer 2008 through summer 2010 audits. At last, for the winter 2011 audit, no participant noted any discrepancies when all were contacted to review their data. We had returned to that great condition where no results were miscalculated (and later corrected), or had “slipped a decimal” or exhibited some other obvious entry error that could have been easily avoided. Unfortunately, for the summer 2011 blind audit, results were AGAIN (!) reported and then later corrected. Happily, for the next five audits (winter 2012 through winter 2014), no participant noted any discrepancies when all were contacted to review their data. Sadly, this improvement in reporting did not extend to the summer 2015 and winter 2016 audits. Results were reported late, or reported and then later corrected.

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 through winter 2016 participants reported only two significant digits for some analytes, thus potentially giving substantial under or over estimates for the comparisons.

CONCLUSION

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

1. Results for particulate carbon and nitrogen were generally consistent between laboratories. Reported concentrations of particulate analytes have usually been similar between laboratories participating in the Blind Audit Program. One laboratory's reported particulate nitrogen concentration was substantially higher than all other laboratories' reported mean concentrations for both audits, although their reported particulate carbon concentration was not significantly different than the mean of the other participants.
2. In contrast to particulate carbon and nitrogen, particulate phosphorus concentrations have shown more variability between participating laboratories in some audit years. For example, there was more particulate phosphorus concentration variability for the winter 2016 audits than in most years, although the concentrations were not particularly different than had been measured in past audits. One laboratory's reported particulate phosphorus concentrations were substantially lower than all other laboratories' reported mean concentrations for both audits. Another laboratory's reported particulate phosphorus concentrations were substantially higher

than all other laboratories' reported mean concentrations for both audits.

3. For all participating laboratories in each audit, there was remarkable consistency between participating laboratories in the measurement of total suspended solids from suspensions of infusorial earth.

4. Most of the chlorophyll *a* results for the summer 2015 and winter 2016 audits displayed the usual close agreement that was remarkable for multi-laboratory comparison of low concentrations of an environmentally transitory compound. Results from no laboratory were consistently different from those of the "consensus" concentrations for both audits.

5. Reported concentrations of dissolved 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 usually execute and report these measurements with accuracy and precision, reporting the appropriate number of significant digits.

6. 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 and orthophosphate. The categories for PASS, 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.

7. There was less variation in reported concentrations of low level ammonium for both these blind audits, in comparison to several previous audits. This probably indicates that inter-laboratory comparisons of any ammonium data prepared from concentrates with resultant concentrations below 0.042 mg N/L could be improving.

8. Care should continue to be taken when completing report forms. For the summer 2015 and winter 2016 blind audits, some results were AGAIN (!) reported with insufficient significant digits. For the summer 2015 and winter 2016 blind audits, some results were reported late, or reported and subsequently corrected. It is hoped that corrections of these lapses have served as reminders of the importance to continuously check many aspects of sample preparation and data management to ensure overall data quality.

Table 1. Participants in Summer 2015 and Winter 2016 Blind Audit Program.

Participant Institution	Point of Contact	Phone	Dissolved	Particulate	Chlorophyll a	DOC	TSS
Old Dominion University, Water Quality Laboratory (ODU)	Suzanne Doughton	757-451-3044	X	X	X	X	X
University of Maryland, Horn Point Laboratory (HPL)	Erica Kiss	410-221-8317	X	X	X	X	X
Virginia Institute of Marine Science, Analytical Service Center (VIMS)	Carol Pollard	804-684-7213	NO L-NH ₄ , PO ₄ W16	PP S15 PCPN W16	X	NO	X
Virginia Division of Consolidated Laboratory Services (DCLS)	Jay Armstrong	804-648-4480 x328	X	X	X	X	X
Maryland Department of Health and Mental Hygiene (DHMH)	Shala Ameli	410-767-6190	X	X	X	X	X
University of Maryland Chesapeake Biological Laboratory (CBL)	Jerry Frank	410-326-7252	X	X	X	X	X
Delaware Department of Natural Resources (DNREC)	Ben Pressly	302-739-9942	X	X	X	X	X
Academy of Natural Science of Philadelphia (ACNAT)	Paul Kiry	215-299-1076	X	X	X	S15	X
Pennsylvania DEP, Bureau of Laboratories (PADEP)	Rebecca Keyes	717-346-8233	HIGH SAMPLES	NO	X	X	X
Massachusetts Water Resources Authority, Central Laboratory (MWRA)	Cara Seaman	617-660-7808	X	X	X	NO	X
Hampton Roads Sanitation District, Central Environmental Laboratory (HRSD)	Chris Bolling	757-460-4217	HIGH SAMPLES	NO	X	S15	X
Ocoquan Watershed Monitoring Lab (OCC)	Dongmei Wang	703-361-5606	X	NO PP	X	X	X
University of Connecticut Center for Environmental Science & Engineering (UCONN)	Chris Perkins	860-486-2668	X	X	X	W16	X
US Geological Survey Indianapolis, IN (IWSC)	Aubrey Bunch	317-600-2783	NO	NO	S15	NO	NO
New Jersey Department of Health (NJDH)	Doug Haltmeier	609-530-2801	NO TDN	NO	NO	X	X
Klamath Tribes Research Station	Craig Spoonmoore	541-783-2149	X	NO	X	NO	X

Table 2. Summary of Mean Concentration and Standard Deviation for Each Group of Analytes in the Summer 2015 and the Winter 2016 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 2015						
Total Dissolved Nitrogen	0.339	0.0178	9	3		
Total Dissolved Nitrogen	0.886	0.0489	9	5		
Total Dissolved Phosphorus	0.0223	0.0031	10	2	1	
Total Dissolved Phosphorus	0.0539	0.0050	14			1
Ammonium	0.026	0.0041	8	4	1	
Ammonium	0.365	0.014	12	1	2	
Nitrate + Nitrite	0.0571	0.0044	9	3	1	
Nitrate + Nitrite	0.773	0.0255	11	3	1	
Orthophosphate	0.0176	0.0021	9	2	2	
Orthophosphate	0.0631	0.0023	10	5		
Dissolved Organic Carbon	2.08	0.16	7	4		
Dissolved Organic Carbon	5.05	0.20	8	2	1	
Particulate Carbon	0.473	0.0255	8	1	1	
Particulate Nitrogen	0.0838	0.0106	7	2	1	
Particulate Phosphorus	0.0119	0.0015	7	2	1	
Total Suspended Solids	42.1	1.69	11	3	1	
Winter 2016						
Total Dissolved Nitrogen	0.163	0.0228	10		2	
Total Dissolved Nitrogen	0.588	0.0378	11	1	2	
Total Dissolved Phosphorus	0.0115	0.0037	10	2		1
Total Dissolved Phosphorus	0.0356	0.0042	12	2		1
Ammonium	0.019	0.0035	9	3		
Ammonium	0.187	0.012	13	1	1	
Nitrate + Nitrite	0.102	0.0077	10	2	1	
Nitrate + Nitrite	0.490	0.0162	12	2	1	
Orthophosphate	0.0089	0.0024	11		1	
Orthophosphate	0.0932	0.0025	9	6		
Dissolved Organic Carbon	4.06	0.15	7	3		
Dissolved Organic Carbon	6.10	0.14	6	4		
Particulate Carbon	1.96	0.140	10		1	
Particulate Nitrogen	0.308	0.0327	9	1	1	
Particulate Phosphorus	0.0168	0.0044	7	1	1	
Total Suspended Solids	66.6	2.79	12	2	1	

Table 3. Summary of Prepared and Reported Concentrations for Each Analyte and Percent Recovery of the Prepared Concentrations by Participating Laboratories

Parameter	Prepared Concentration mg/L	Reported Concentration Range mg/L	Number of Laboratories		
			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 2015					
Total Dissolved Nitrogen	0.338	0.303-0.372	8	2	2
Total Dissolved Nitrogen	0.888	0.810-0.970	12	2	
Total Dissolved Phosphorus	0.0210	0.0180-0.0303	8	3	2
Total Dissolved Phosphorus	0.0520	0.0490-0.0700	12	1	1
Ammonium	0.025	0.020-0.035	8	2	3
Ammonium	0.361	0.337-0.395	15		
Nitrate + Nitrite	0.0561	0.0510-0.0680	11	1	1
Nitrate + Nitrite	0.771	0.730-0.832	15		
Orthophosphate	0.0163	0.0132-0.0220	9	1	3
Orthophosphate	0.0609	0.0596-0.0665	15		
Dissolved Organic Carbon	2.00	1.85-2.37	8	3	
Dissolved Organic Carbon	5.00	4.76-5.49	11		
Total Suspended Solids	44.7	39.0-46.1	13	2	
Winter 2016					
Total Dissolved Nitrogen	0.169	0.112-0.211	11		
Total Dissolved Nitrogen	0.592	0.510-0.680	12	1	
Total Dissolved Phosphorus	0.010	0.0070-0.0230	6	4	3
Total Dissolved Phosphorus	0.033	0.030-0.049	10	4	1
Ammonium	0.017	0.012-0.024	4	2	6
Ammonium	0.191	0.151-0.207	14		1
Nitrate + Nitrite	0.0981	0.0902-0.1223	11	1	1
Nitrate + Nitrite	0.490	0.449-0.523	15		
Orthophosphate	0.0074	0.0070-0.0160	6	2	4
Orthophosphate	0.0928	0.0888-0.0980	15		
Dissolved Organic Carbon	4.00	3.76-4.27	10		
Dissolved Organic Carbon	6.00	5.93-6.28	10		
Total Suspended Solids	74.5	60.1-70.0	9	6	

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

Appendix 1 Summer 2015 and Winter 2016 Reported Concentrations, Prepared Concentrations and Recoveries**University of Connecticut Center for Environmental Science and Engineering (UCONN)**

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.345		0.338	102.1		0.163		0.169	96.4
TDN (mg N/L)	0.917		0.888	103.3		0.582		0.592	98.3
TDP (mg P/L)	0.0220		0.021	104.8		0.0120		0.01	120.0
TDP (mg P/L)	0.0510		0.052	98.1		0.0340		0.033	103.0
NH4 (mg N/L)	0.030		0.025	120.0		0.022		0.017	129.4
NH4 (mg N/L)	0.366		0.361	101.4		0.186		0.191	97.4
NO23 (mg N/L)	0.06		0.0561	107.0		0.1		0.0981	101.9
NO23 (mg N/L)	0.832	W	0.7713	107.9		0.498		0.4902	101.6
PO4 (mg P/L)	0.02		0.0163	122.7		0.016	W	0.0074	216.2
PO4 (mg P/L)	0.061		0.0609	100.2		0.089		0.0928	95.9
PC (mg C/L)	0.4650		NA	NA		2.0080		NA	NA
PN (mg N/L)	0.0720		NA	NA		0.3115		NA	NA
PP (mg P/L)	0.0100		NA	NA		0.0155		NA	NA
CHL (ug/L)	11.3		NA	NA		27.8		NA	NA
DOC (mg C/L)	*		2.00	*		4.08		4.00	102.0
DOC (mg C/L)	*		5.00	*		6.28		6.00	104.7
TSS (mg/L)	42.5		44.7	95.1		64.8		74.5	87.0

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Academy of Natural Science of Philadelphia (ACNAT)

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.341		0.338	100.9		0.175		0.169	103.6
TDN (mg N/L)	0.871		0.888	98.1		0.534		0.592	90.2
TDP (mg P/L)	0.0202		0.021	96.2		0.0077		0.01	77.4
TDP (mg P/L)	0.0519		0.052	99.8		0.0336		0.033	101.8
NH4 (mg N/L)	0.026		0.025	102.0		0.014		0.017	80.6
NH4 (mg N/L)	0.359		0.361	99.4		0.189		0.191	99.0
NO23 (mg N/L)	0.0524		0.0561	93.4		0.0902		0.0981	91.9
NO23 (mg N/L)	0.749		0.7713	97.1		0.47		0.4902	95.9
PO4 (mg P/L)	0.0165		0.0163	101.2		0.0082		0.0074	110.8
PO4 (mg P/L)	0.0608		0.0609	99.8		0.0918		0.0928	98.9
PC (mg C/L)	0.4100	W	NA	NA		1.5700	W	NA	NA
PN (mg N/L)	0.0684		NA	NA		0.2680		NA	NA
PP (mg P/L)	0.0122		NA	NA		0.0173		NA	NA
CHL (ug/L)	8.4		NA	NA		30.9		NA	NA
DOC (mg C/L)	1.98		2.00	*		*		4.00	*
DOC (mg C/L)	5.02		5.00	*		*		6.00	*
TSS (mg/L)	39.6		44.7	88.6		66.2		74.5	88.9

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Appendix 1 Cont'. Summer 2015 and Winter 2016 Reported Concentrations, Prepared Concentrations and Recoveries**Virginia Institute of Marine Science, Analytical Service Center (VIMS)**

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.324		0.338	95.9		0.112	W	0.169	66.1
TDN (mg N/L)	0.965		0.888	108.7		0.610		0.592	103.1
TDP (mg P/L)	0.0259		0.021	123.3		0.0111		0.01	111.0
TDP (mg P/L)	0.0552		0.052	106.2		0.0344		0.033	104.2
NH4 (mg N/L)	0.020		0.025	79.2		*		0.017	*
NH4 (mg N/L)	0.356		0.361	98.7		0.182		0.191	95.1
NO23 (mg N/L)	0.051		0.0561	90.9		0.1223	W	0.0981	124.7
NO23 (mg N/L)	0.7716		0.7713	100.0		0.5062		0.4902	103.3
PO4 (mg P/L)	0.0132	W	0.0163	81.0		*		0.0074	*
PO4 (mg P/L)	0.0644		0.0609	105.7		0.0916		0.0928	98.7
PC (mg C/L)	*		NA	NA		2.0550		NA	NA
PN (mg N/L)	*		NA	NA		0.2945		NA	NA
PP (mg P/L)	0.0120		NA	NA		*		NA	NA
CHL (ug/L)	9.17		NA	NA		31.05		NA	NA
DOC (mg C/L)	*		2.00	*		*		4.00	*
DOC (mg C/L)	*		5.00	*		*		6.00	*
TSS (mg/L)	41.8		44.7	93.5		67.3		74.5	90.3

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Hampton Roads Sanitation District, Central Environmental Laboratory (HRSRD)

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	*		0.338	*		*		0.169	*
TDN (mg N/L)	0.970		0.888	109.2		0.510	W	0.592	86.1
TDP (mg P/L)	*		0.021	*		*		0.01	*
TDP (mg P/L)	0.0500		0.052	96.2		0.0310		0.033	93.9
NH4 (mg N/L)	*		0.025	*		*		0.017	*
NH4 (mg N/L)	0.370		0.361	102.5		0.190		0.191	99.5
NO23 (mg N/L)	*		0.0561	*		*		0.0981	*
NO23 (mg N/L)	0.78		0.7713	101.1		0.49		0.4902	100.0
PO4 (mg P/L)	*		0.0163	*		*		0.0074	*
PO4 (mg P/L)	0.06		0.0609	98.5		0.098		0.0928	105.6
PC (mg C/L)	*		NA	NA		*		NA	NA
PN (mg N/L)	*		NA	NA		*		NA	NA
PP (mg P/L)	*		NA	NA		*		NA	NA
CHL (ug/L)	8.9		NA	NA		22.2		NA	NA
DOC (mg C/L)	2.37		2.00	118.5		*		4.00	*
DOC (mg C/L)	4.76		5.00	95.2		*		6.00	*
TSS (mg/L)	42.6		44.7	95.3		64.5		74.5	86.6

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Appendix 1 Cont'. Summer 2015 and Winter 2016 Reported Concentrations, Prepared Concentrations and Recoveries**University of Maryland, Horn Point Laboratory (HPL)**

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.343		0.338	101.5		0.147		0.169	87.0
TDN (mg N/L)	0.889		0.888	100.1		0.578		0.592	97.6
TDP (mg P/L)	0.0224		0.021	106.7		0.0110		0.01	110.0
TDP (mg P/L)	0.0572		0.052	110.0		0.0381		0.033	115.5
NH4 (mg N/L)	0.020		0.025	81.6		0.018		0.017	107.1
NH4 (mg N/L)	0.361		0.361	100.0		0.189		0.191	99.0
NO23 (mg N/L)	0.0584		0.0561	104.1		0.1		0.0981	101.9
NO23 (mg N/L)	0.778		0.7713	100.9		0.498		0.4902	101.6
PO4 (mg P/L)	0.0166		0.0163	101.8		0.00742		0.0074	100.3
PO4 (mg P/L)	0.0639		0.0609	104.9		0.0942		0.0928	101.5
PC (mg C/L)	0.4920		NA	NA		1.9850		NA	NA
PN (mg N/L)	0.0828		NA	NA		0.2875		NA	NA
PP (mg P/L)	0.0155	W	NA	NA		0.0235		NA	NA
CHL (ug/L)	5.66		NA	NA		17.55		NA	NA
DOC (mg C/L)	2.00		2.00	100.0		3.95		4.00	98.8
DOC (mg C/L)	4.93		5.00	98.6		5.93		6.00	98.8
TSS (mg/L)	41.4		44.7	92.6		70		74.5	94.0

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Delaware Department of Natural Resources (DNREC)

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.325		0.338	96.2		0.211	W	0.169	124.9
TDN (mg N/L)	0.828		0.888	93.2		0.615		0.592	103.9
TDP (mg P/L)	0.0240		0.021	114.3		0.0070		0.01	70.0
TDP (mg P/L)	0.0540		0.052	103.8		0.0300		0.033	90.9
NH4 (mg N/L)	0.032		0.025	128.0		0.024		0.017	142.9
NH4 (mg N/L)	0.355		0.361	98.4		0.183		0.191	95.8
NO23 (mg N/L)	0.068	W	0.0561	121.2		0.101		0.0981	103.0
NO23 (mg N/L)	0.73		0.7713	94.6		0.449	W	0.4902	91.6
PO4 (mg P/L)	0.02		0.0163	122.7		0.01		0.0074	135.1
PO4 (mg P/L)	0.066		0.0609	108.4		0.093		0.0928	100.2
PC (mg C/L)	0.4983		NA	NA		2.0800		NA	NA
PN (mg N/L)	0.0799		NA	NA		0.3100		NA	NA
PP (mg P/L)	0.0097		NA	NA		0.0063	W	NA	NA
CHL (ug/L)	8.212		NA	NA		22.35		NA	NA
DOC (mg C/L)	2.21		2.00	110.5		4.20		4.00	105.0
DOC (mg C/L)	5.49	W	5.00	109.8		6.24		6.00	104.0
TSS (mg/L)	42.67		44.7	95.5		68.9		74.5	92.5

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Appendix 1 Cont'. Summer 2015 and Winter 2016 Reported Concentrations, Prepared Concentrations and Recoveries**Division of Consolidated Laboratory Services (DCLS)**

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.326		0.338	96.4		0.170		0.169	100.6
TDN (mg N/L)	0.850		0.888	95.7		0.605		0.592	102.2
TDP (mg P/L)	0.0180		0.021	85.7		0.0110		0.01	110.0
TDP (mg P/L)	0.0490		0.052	94.2		0.0360		0.033	109.1
NH4 (mg N/L)	0.026		0.025	104.0		0.012		0.017	70.6
NH4 (mg N/L)	0.358		0.361	99.2		0.178		0.191	93.2
NO23 (mg N/L)	0.057		0.0561	101.6		0.102		0.0981	104.0
NO23 (mg N/L)	0.81		0.7713	105.0		0.496		0.4902	101.2
PO4 (mg P/L)	0.018		0.0163	110.4		0.008		0.0074	108.1
PO4 (mg P/L)	0.064		0.0609	105.1		0.096		0.0928	103.4
PC (mg C/L)	0.4810		NA	NA		1.9750		NA	NA
PN (mg N/L)	0.0840		NA	NA		0.2835		NA	NA
PP (mg P/L)	0.0113		NA	NA		0.0147		NA	NA
CHL (ug/L)	9.83		NA	NA		26.55		NA	NA
DOC (mg C/L)	1.99		2.00	99.5		3.96		4.00	99.0
DOC (mg C/L)	5.07		5.00	101.4		6.03		6.00	100.5
TSS (mg/L)	41.0		44.7	91.7		68		74.5	91.3

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

University of Maryland, Chesapeake Biological Laboratory (CBL)

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.372		0.338	110.1		0.178		0.169	105.3
TDN (mg N/L)	0.951		0.888	107.1		0.596		0.592	100.7
TDP (mg P/L)	0.0195		0.021	92.9		0.0109		0.01	109.0
TDP (mg P/L)	0.0515		0.052	99.0		0.0346		0.033	104.8
NH4 (mg N/L)	0.025		0.025	100.0		0.021		0.017	123.5
NH4 (mg N/L)	0.395	W	0.361	109.4		0.207		0.191	108.4
NO23 (mg N/L)	0.0559		0.0561	99.6		0.113		0.0981	115.2
NO23 (mg N/L)	0.786		0.7713	101.9		0.523		0.4902	106.7
PO4 (mg P/L)	0.0165		0.0163	101.2		0.0075		0.0074	101.4
PO4 (mg P/L)	0.0652		0.0609	107.1		0.0939		0.0928	101.2
PC (mg C/L)	0.5000		NA	NA		2.0100		NA	NA
PN (mg N/L)	0.0812		NA	NA		0.2900		NA	NA
PP (mg P/L)	0.0113		NA	NA		0.0195		NA	NA
CHL (ug/L)	7.16		NA	NA		26.17		NA	NA
DOC (mg C/L)	2.03		2.00	101.5		4.14		4.00	103.5
DOC (mg C/L)	5.03		5.00	100.6		6.16		6.00	102.7
TSS (mg/L)	40.6		44.7	90.8		66.1		74.5	88.7

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Appendix 1 Cont'. Summer 2015 and Winter 2016 Reported Concentrations, Prepared Concentrations and Recoveries**Virginia Polytechnic Institute, Occoquan Watershed Monitoring Laboratory (OCC)**

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.365		0.338	108.0		0.158		0.169	93.5
TDN (mg N/L)	0.870		0.888	98.0		0.603		0.592	101.9
TDP (mg P/L)	0.0200		0.021	95.2		0.0230	F	0.01	230.0
TDP (mg P/L)	0.0530		0.052	101.9		0.0490	F	0.033	148.5
NH4 (mg N/L)	0.035	W	0.025	140.0		0.022		0.017	129.4
NH4 (mg N/L)	0.368		0.361	101.9		0.197		0.191	103.1
NO23 (mg N/L)	0.054		0.0561	96.3		0.097		0.0981	98.9
NO23 (mg N/L)	0.753		0.7713	97.6		0.497		0.4902	101.4
PO4 (mg P/L)	0.022	W	0.0163	135.0		0.01		0.0074	135.1
PO4 (mg P/L)	0.066		0.0609	108.4		0.093		0.0928	100.2
PC (mg C/L)	0.4610		NA	NA		1.8200		NA	NA
PN (mg N/L)	0.1080	W	NA	NA		0.3960	W	NA	NA
PP (mg P/L)	*		NA	NA		*		NA	NA
CHL (ug/L)	12.5		NA	NA		16.2		NA	NA
DOC (mg C/L)	2.33		2.00	116.4		4.27		4.00	106.8
DOC (mg C/L)	5.01		5.00	100.1		6.25		6.00	104.2
TSS (mg/L)	43.5		44.7	97.3		67.9		74.5	91.1

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Maryland Department of Health and Mental Hygiene (DHMH)

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.335		0.338	99.1		0.147		0.169	87.0
TDN (mg N/L)	0.852		0.888	95.9		0.588		0.592	99.3
TDP (mg P/L)	0.0209		0.021	99.5		0.0108		0.01	108.0
TDP (mg P/L)	0.0492		0.052	94.6		0.0338		0.033	102.4
NH4 (mg N/L)	0.026		0.025	102.8		0.020		0.017	115.3
NH4 (mg N/L)	0.365		0.361	101.1		0.196		0.191	102.6
NO23 (mg N/L)	0.0559		0.0561	99.6		0.0979		0.0981	99.8
NO23 (mg N/L)	0.748		0.7713	97.0		0.484		0.4902	98.7
PO4 (mg P/L)	0.0177		0.0163	108.6		0.00791		0.0074	106.9
PO4 (mg P/L)	0.0646		0.0609	106.1		0.0888		0.0928	95.7
PC (mg C/L)	0.4560		NA	NA		2.0185		NA	NA
PN (mg N/L)	0.0937		NA	NA		0.3210		NA	NA
PP (mg P/L)	0.0125		NA	NA		0.0171		NA	NA
CHL (ug/L)	7.92		NA	NA		28.1		NA	NA
DOC (mg C/L)	2.10		2.00	105.1		4.18		4.00	104.5
DOC (mg C/L)	5.00		5.00	100.0		6.09		6.00	101.5
TSS (mg/L)	41.5		44.7	92.8		69.1		74.5	92.8

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Appendix 1 Cont'. Summer 2015 and Winter 2016 Reported Concentrations, Prepared Concentrations and Recoveries**Massachusetts Water Resource Authority, Central Laboratory (MWRA)**

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.342		0.338	101.2		0.178		0.169	105.3
TDN (mg N/L)	0.906		0.888	102.0		0.584		0.592	98.6
TDP (mg P/L)	0.0239		0.021	113.8		0.0115		0.01	115.0
TDP (mg P/L)	0.0582		0.052	111.9		0.0372		0.033	112.7
NH4 (mg N/L)	0.024		0.025	96.4		0.017		0.017	99.4
NH4 (mg N/L)	0.395	W	0.361	109.4		0.192		0.191	100.5
NO23 (mg N/L)	0.0541		0.0561	96.4		0.103		0.0981	105.0
NO23 (mg N/L)	0.744		0.7713	96.5		0.501		0.4902	102.2
PO4 (mg P/L)	0.018		0.0163	110.4		0.00874		0.0074	118.1
PO4 (mg P/L)	0.0665		0.0609	109.2		0.0952		0.0928	102.6
PC (mg C/L)	0.4800		NA	NA		2.0250		NA	NA
PN (mg N/L)	0.0794		NA	NA		0.3330		NA	NA
PP (mg P/L)	0.0119		NA	NA		0.0192		NA	NA
CHL (ug/L)	10.09		NA	NA		35.05		NA	NA
DOC (mg C/L)	*		2.00	*		*		4.00	*
DOC (mg C/L)	*		5.00	*		*		6.00	*
TSS (mg/L)	46.1	W	44.7	103.1		60.1	W	74.5	80.7

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Old Dominion University, Water Quality Laboratory (ODU)

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.348		0.338	103.0		0.166		0.169	98.2
TDN (mg N/L)	0.880		0.888	99.1		0.587		0.592	99.2
TDP (mg P/L)	0.0209		0.021	99.5		0.0094		0.01	94.0
TDP (mg P/L)	0.0533		0.052	102.5		0.0339		0.033	102.7
NH4 (mg N/L)	0.024		0.025	96.4		0.019		0.017	110.0
NH4 (mg N/L)	0.368		0.361	101.9		0.193		0.191	101.0
NO23 (mg N/L)	0.0567		0.0561	101.1		0.1		0.0981	101.9
NO23 (mg N/L)	0.781		0.7713	101.3		0.491		0.4902	100.2
PO4 (mg P/L)	0.0174		0.0163	106.7		0.007		0.0074	94.6
PO4 (mg P/L)	0.0619		0.0609	101.6		0.0936		0.0928	100.9
PC (mg C/L)	0.4875		NA	NA		2.0500		NA	NA
PN (mg N/L)	0.0890		NA	NA		0.2985		NA	NA
PP (mg P/L)	0.0128		NA	NA		0.0182		NA	NA
CHL (ug/L)	9.83		NA	NA		29.15		NA	NA
DOC (mg C/L)	1.85		2.00	92.5		3.88		4.00	97.0
DOC (mg C/L)	4.96		5.00	99.2		5.93		6.00	98.8
TSS (mg/L)	43.9		44.7	98.2		68.6		74.5	92.1

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Appendix 1 Cont'. Summer 2015 and Winter 2016 Reported Concentrations, Prepared Concentrations and Recoveries**Pennsylvania Department of Environmental Protection, Bureau of Laboratories (PADEP)**

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration		Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration		Winter 2016 Percent Recovered	
TDN (mg N/L)	*		0.338		*		*		0.169		*	
TDN (mg N/L)	0.810		0.888		91.2		0.680	W	0.592		114.9	
TDP (mg P/L)	*		0.021		*		*		0.01		*	
TDP (mg P/L)	0.0700	F	0.052		134.6		0.0340		0.033		103.0	
NH4 (mg N/L)	*		0.025		*		*		0.017		*	
NH4 (mg N/L)	0.337		0.361		93.4		0.151	W	0.191		79.1	
NO23 (mg N/L)	*		0.0561		*		*		0.0981		*	
NO23 (mg N/L)	0.77		0.7713		99.8		0.48		0.4902		97.9	
PO4 (mg P/L)	*		0.0163		*		*		0.0074		*	
PO4 (mg P/L)	0.061		0.0609		100.2		0.094		0.0928		101.3	
PC (mg C/L)	*		NA		NA		*		NA		NA	
PN (mg N/L)	*		NA		NA		*		NA		NA	
PP (mg P/L)	*		NA		NA		*		NA		NA	
CHL (ug/L)	11.58		NA		NA		29.25		NA		NA	
DOC (mg C/L)	1.86		2.00		93.0		3.76		4.00		94.0	
DOC (mg C/L)	4.86		5.00		97.2		5.88		6.00		98.0	
TSS (mg/L)	39		44.7		87.2		61		74.5		81.9	

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

New Jersey Department of Health (NJDH)

	Summer 2015 Reported Concentration		Summer 2015 Prepared Concentration		Summer 2015 Percent Recovered		Winter 2016 Reported Concentration		Winter 2016 Prepared Concentration		Winter 2016 Percent Recovered	
TDN (mg N/L)	*		0.338		*		*		0.169		*	
TDN (mg N/L)	*		0.888		*		*		0.592		*	
TDP (mg P/L)	0.0303	W	0.021		144.3		0.0123		0.01		123.0	
TDP (mg P/L)	0.0528		0.052		101.5		0.0373		0.033		113.0	
NH4 (mg N/L)	0.024		0.025		95.6		0.017		0.017		97.1	
NH4 (mg N/L)	0.360		0.361		99.7		0.196		0.191		102.6	
NO23 (mg N/L)	0.0635		0.0561		113.2		0.104		0.0981		106.0	
NO23 (mg N/L)	0.785		0.7713		101.8		0.488		0.4902		99.6	
PO4 (mg P/L)	0.0168		0.0163		103.1		0.0091		0.0074		123.0	
PO4 (mg P/L)	0.0596		0.0609		97.9		0.09		0.0928		97.0	
PC (mg C/L)	*		NA		NA		*		NA		NA	
PN (mg N/L)	*		NA		NA		*		NA		NA	
PP (mg P/L)	*		NA		NA		*		NA		NA	
CHL (ug/L)	*		NA		NA		*		NA		NA	
DOC (mg C/L)	2.11		2.00		105.5		4.14		4.00		103.5	
DOC (mg C/L)	5.38		5.00		107.6		6.20		6.00		103.4	
TSS (mg/L)	42.1		44.7		94.2		67.4		74.5		90.5	

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Appendix 1 Cont'. Summer 2015 and Winter 2016 Reported Concentrations, Prepared Concentrations and Recoveries**USGS - Indiana Water Science Center (IWSC)**

	Summer 2015 Reported Concentration	Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered	Winter 2016 Reported Concentration	Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	*	0.338	*	*	0.169	*
TDN (mg N/L)	*	0.888	*	*	0.592	*
TDP (mg P/L)	*	0.021	*	*	0.01	*
TDP (mg P/L)	*	0.052	*	*	0.033	*
NH4 (mg N/L)	*	0.025	*	*	0.017	*
NH4 (mg N/L)	*	0.361	*	*	0.191	*
NO23 (mg N/L)	*	0.0561	*	*	0.0981	*
NO23 (mg N/L)	*	0.7713	*	*	0.4902	*
PO4 (mg P/L)	*	0.0163	*	*	0.0074	*
PO4 (mg P/L)	*	0.0609	*	*	0.0928	*
PC (mg C/L)	*	NA	NA	*	NA	NA
PN (mg N/L)	*	NA	NA	*	NA	NA
PP (mg P/L)	*	NA	NA	*	NA	NA
CHL (ug/L)	9.46	NA	NA	*	NA	NA
DOC (mg C/L)	*	2.00	*	*	4.00	*
DOC (mg C/L)	*	5.00	*	*	6.00	*
TSS (mg/L)	*	44.7	*	*	74.5	*

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Klamath Tribe's Research Station (KLAM)

	Summer 2015 Reported Concentration	Summer 2015 Prepared Concentration	Summer 2015 Percent Recovered	Winter 2016 Reported Concentration	Winter 2016 Prepared Concentration	Winter 2016 Percent Recovered
TDN (mg N/L)	0.303	0.338	89.6	0.152	0.169	89.9
TDN (mg N/L)	0.837	0.888	94.3	0.563	0.592	95.1
TDP (mg P/L)	0.0220	0.021	104.8	0.0120	0.01	120.0
TDP (mg P/L)	0.0520	0.052	100.0	0.0370	0.033	112.1
NH4 (mg N/L)	0.025	0.025	100.0	0.022	0.017	129.4
NH4 (mg N/L)	0.356	0.361	98.6	0.183	0.191	95.8
NO23 (mg N/L)	0.056	0.0561	99.8	0.096	0.0981	97.9
NO23 (mg N/L)	0.784	0.7713	101.6	0.484	0.4902	98.7
PO4 (mg P/L)	0.016	0.0163	98.2	0.007	0.0074	94.6
PO4 (mg P/L)	0.061	0.0609	100.2	0.096	0.0928	103.4
PC (mg C/L)	*	NA	NA	*	NA	NA
PN (mg N/L)	*	NA	NA	*	NA	NA
PP (mg P/L)	*	NA	NA	*	NA	NA
CHL (ug/L)	10.3	NA	NA	33.9	NA	NA
DOC (mg C/L)	*	2.00	*	*	4.00	*
DOC (mg C/L)	*	5.00	*	*	6.00	*
TSS (mg/L)	42.8	44.7	95.7	68.5	74.5	91.9

* No sample sent to participant - sample not requested, parameter or concentration range not routine

"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

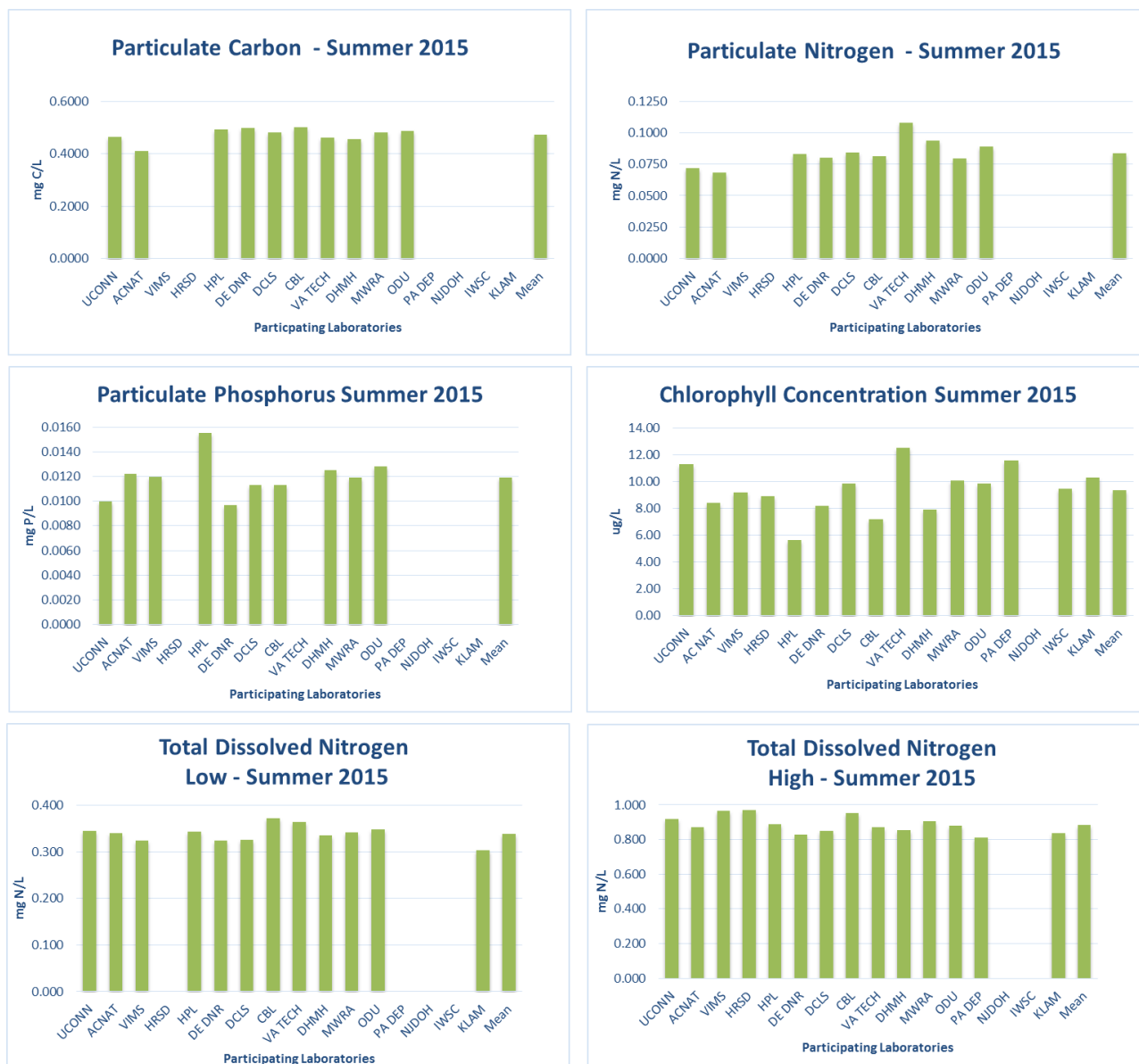


Figure 1. Particulate carbon, nitrogen and phosphorus; chlorophyll a, and total dissolved nitrogen. Summer 2015



Figure 2. Total dissolved phosphorus; dissolved organic carbon, and ammonium. Summer 2015



Figure 3. Nitrite plus nitrate, orthophosphate, and total suspended solids. Summer 2015

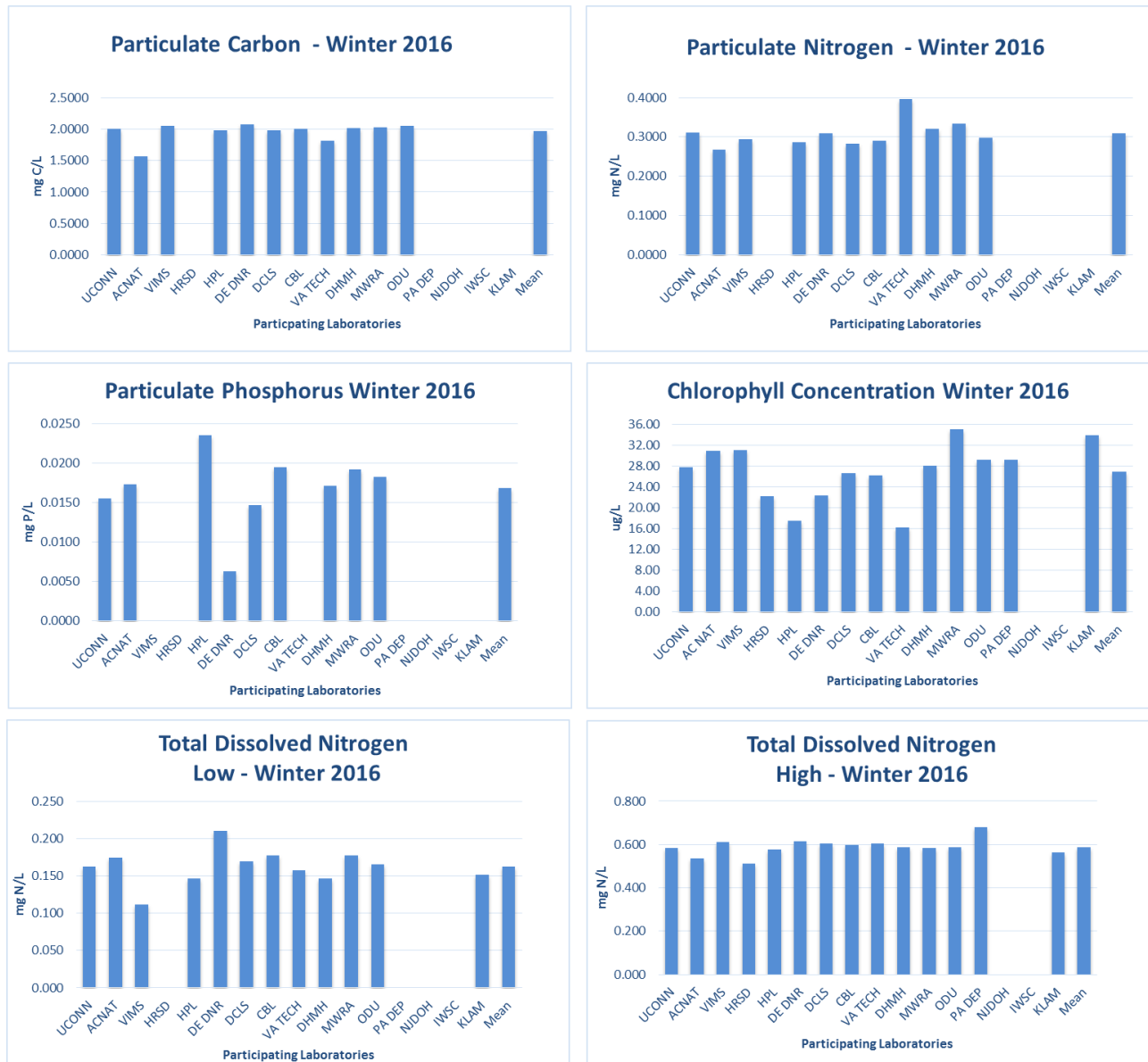


Figure 4. Particulate carbon, nitrogen and phosphorus; chlorophyll a, and total dissolved nitrogen. Winter 2016.



Figure 5. Total dissolved phosphorus; dissolved organic carbon, and ammonium. Winter 2016.



Figure 6. Nitrite plus nitrate, orthophosphate, and total suspended solids. Winter 2016.