FINAL REPORT

CHESAPEAKE BAY PROGRAM BLIND AUDIT

Fiscal Year 2018 Final Report

PREPARED FOR:

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Resource Assessment Administration
Water and Habitat Quality Program
Annapolis, MD 21401

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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, its tributaries, or similar systems. The concentrations of these samples, which are unknown to the recipient analysts, are compared to their prepared concentrations, or on the case of particulate samples, the range of values reported.

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 twentieth 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 4 December 2017 and 29 May 2018. 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 orthophosphate). 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 vacuum filtered (</= 10 in Hg, or 5 psi) from the batch sample with care taken to shake the batch before each filtration to ensure homogeneity. 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 fall 2017 and spring 2018 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

<u>Total Dissolved Nitrogen:</u> Results from both the fall 2017 and spring 2018 audits were about as in past audits, with one laboratory reporting a concentration that was not close to other participants.

<u>Total Dissolved Phosphorus:</u> Most reported concentrations for both fall 2017 and spring 2018 samples were consistently close to other laboratories' reported concentrations except for one participant in each audit.

<u>Ammonium:</u> All reported concentrations for both audits were consistently close to other laboratories' reported concentrations. Analysis of low level samples for fall 2017 provided a mean concentration of 0.021 mg N/L compared to the prepared concentration of 0.021 mg N/L.

<u>Nitrate + Nitrite:</u> Particularly good agreement was found among most laboratories for low concentrations, resulting in a mean concentration of 0.0948 mg N/L for spring 2018 compared to the prepared concentration of 0.0981 mg N/L.

Orthophosphate: Good agreement was found among all laboratories for both low level and high level concentrations for both fall 2017 and spring 2018 audits.

<u>Dissolved Organic Carbon:</u> Particularly good agreement was found among all laboratories for low and high concentrations for both 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.

<u>Particulate Carbon:</u> Among laboratory agreement was close for the fall 2017 and spring 2018 audits with coefficients of variation of only 5-9%.

<u>Particulate Nitrogen:</u> Results for particulate nitrogen followed the same pattern as particulate carbon for both audits with coefficients of variation 7-10%.

<u>Particulate Phosphorus:</u> Particulate phosphorus concentrations showed some variability between the participating laboratories with one laboratory reporting substantially a different

concentration from the other participants for the fall 2017 audit but not in the spring 2018 audit.

<u>Chlorophyll a:</u> Most chlorophyll a results for the fall 2017 and spring 2018 audits displayed the usual close agreement that was remarkable for multi-laboratory comparison of low concentrations of an environmentally transitory compound. The coefficients of variation were 16% for the fall/fall 2016 samples and 22% for the spring/spring 2017 samples.

<u>Total Suspended Solids:</u> 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 fall 2017 sample, 25.0 mg/L was prepared and for the spring 2018 sample, 67.4 mg/L was prepared, obtaining coefficients of variation only 8-10% for each audit.

DISCUSSION

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

<u>Variation Associated With An Analytical Method:</u> As we have noted in previous Blind Audit Reports, analytical variability is associated with any quantitative determination. The method detection limit (MDL) 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. <u>Any</u> 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 +/- 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 fall 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 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 fall 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 spring 2016 blind audit of 0.014 mg N/L prepared for ammonium has a PASS category (+/-10%) of only 0.013 - 0.015 mg N/L. For the spring 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 +/-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, spring 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 fall 2012 through fall 2014 low level ammonium samples than in audits of fall 2011 and spring 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 fall 2015 concentration of 0.025 mg NH4-N/L; 19% for spring 2016 concentration of 0.014. For the spring 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 (Fall 2006) and 39% for 0.036 mg NH4-N/L (Spring 2007). This indicates that inter-laboratory comparisons of any ammonium data prepared by most laboratories from concentrates below 0.042 mg N/L may be improving.

There were sixteen 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 10% of the mean reported concentrations for particulate carbon and nitrogen for the fall 2017 and spring 2018 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 spring 1998 was approximately 0.45 mg C/L, and in spring 2013 was approximately 2.35 mg C/L. Particulate phosphorus in spring 2014 was 0.0091 mg P/L and in spring 1999 was 0.0529 mg P/L.

<u>Reporting Data Accurately:</u> 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 fall 2007 and spring 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. This improvement in reporting did not extend to the fall 2008 through fall 2010 audits. At last, for the spring 2011 audit, no participant noted any discrepancies when all were contacted to review their data. We had returned to that 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 fall 2011 blind audit, results were reported and then later corrected. The next five audits (spring 2012 through spring 2014), no participant noted any discrepancies when all were contacted to review their data. This improvement in reporting did not extend to the next six audits; fall 2015 and spring 2016, fall 2016 and spring 2017, fall 2017 and spring 2018. 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 spring 2016 participants reported only two significant digits for some analytes, thus potentially giving substantial under or over estimates for the comparisons.

CONCLUSION

Now that forty-one 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. This was true again this year.
- 2. In contrast to particulate carbon and nitrogen, particulate phosphorus concentrations have shown more variability between participating laboratories in some audit years. This year all participants reported particulate phosphorus concentrations were consistent with each other, with spring 2018 showing better agreement across the cohort of participants.
- 3. For all participating laboratories in both audits, 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 fall 2017 and spring 2018 audits displayed 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 <u>both</u> 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 total dissolved phosphorus, 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 were nineteen instances in which reported data for dissolved constituents or total suspended solids fell in the WARN or FAIL category based on the standard deviation of all participants' data AND the percent recovery of the prepared analyte.
- 8. Care should continue to be taken when completing report forms. For the fall 2017 and spring 2018 blind audits, a few results were reported with insufficient significant digits. For the fall 2017 and spring 2018 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 Fall 2017 and Spring 2018 Blind Audit Program.

| Participant Institution | Point of Contact | Phone | Email |
|--|----------------------------|----------------------|--|
| Old Dominion University, Water Quality Laboratory (ODU) | Suzanne Doughton | 757-451-3044 | sdoughte@odu.edu |
| University of Maryland, Horn Point Laboratory (HPL) | Erica Kiss | 410-221-8317 | ekiss@umces.edu |
| Virginia Institute of Marine Science, Analytical Service Center (VIMS) | Carol Pollard | 804-684-7213 | pollard@vims.edu |
| Virginia Division of Consolidated Laboratory Services (DCLS) | Jay Armstrong | 804-648-4480 x328 | jay.armstrong@dgs.virginia.gov |
| Maryland Department of Health (MDH) | Shala Ameli | 410-767-6190 | shahla.ameli@maryland.gov |
| University of Maryland Chesapeake Biological Laboratory (CBL) | Jerry Frank | 410-326-7252 | frank@umces.edu |
| Delaware Department of Natural Resources (DNREC) | Cathy Sim Kathy Knowles | 302-739-9276 | catherine.sim@state.de.us kathy.knowles@state.de.us |
| Academy of Natural Science of Philadelphia (ACNAT) | Paul Kiry | 215-299-1076 | kiry@ansp.org |
| Pennsylvania DEP, Bureau of Laboratories (PADEP) | Jayne Hogue | 717-346-8233 | jahogue@pa.gov |
| Massachusetts Water Resources Authority, Central Laboratory (MWRA) | Jennifer Constantino | 617-660-7808 | jennifer.constantino@mwra.com |
| Hampton Roads Sanitation District, Central Environmental Laboratory (HRSD) | Reggie Morgan | 757-460-4210 | rmorgan@hrsd.com |
| Occoquan Watershed Monitoring Lab (OWML) | Dongmei Wang | 703-361-5606 | dongmw4@vt.edu |
| University of Connecticut Center for Environmental Science & Engineering (UCONN) | Chris Perkins | 860-486-2668 | christopher.perkins@uconn.edu |
| New Jersey Department of Health (NJDH) | Doug Haltmeier | 609-530-2801 | douglas.haltmeier@doh.nj.gov |
| Sprague River Water Quality Laboratory (SRWQL) | Ben Harris | 541-827-5231 | ben.harris@klamathtribes.com |
| Microbac Laboratories Inc. (MICRO) | Curtis Read | 804-353-1999 | curtis.read@microbac.com |
| University of Maryland Appalachian Laboratory (AL) | Katie Kline | 301-689-7122 | kkline@al.umces.edu |

Table 2. Summary of Mean Concentration and Standard Deviation for Each Group of Analytes in the Fall 2017 and the Spring 2018 Blind Audit, Including Distribution of Reported Concentrations from the Mean.

| Parameter | | | Number of Laboratories | | | | |
|----------------------------|--------|-----------------|-------------------------------|------|------|------|--|
| | Concer | tration in mg/L | Standard Deviations from Mean | | | | |
| | | | <1 | 1-2 | 2-3 | >3 | |
| | Mean | S.D. | PASS | PASS | WARN | FAIL | |
| Fall 2017 | | | | | | | |
| Particulate Carbon | 0.922 | 0.0464 | 9 | 1 | 1 | | |
| Particulate Nitrogen | 0.133 | 0.0097 | 10 | | 1 | | |
| Particulate Phosphorus | 0.0190 | 0.0036 | 8 | 2 | 1 | | |
| Total Suspended Solids | 23.1 | 1.79 | 10 | 6 | | | |
| Total Dissolved Nitrogen | 0.356 | 0.0429 | 9 | 3 | 1 | | |
| Total Dissolved Nitrogen | 0697 | 0.0337 | 12 | 2 | 1 | | |
| Total Dissolved Phosphorus | 0.0271 | 0.00340 | 6 | 8 | | | |
| Total Dissolved Phosphorus | 0.0557 | 0.00736 | 11 | 4 | 1 | | |
| Dissolved Organic Carbon | 3.09 | 0.150 | 7 | 2 | 1 | | |
| Dissolved Organic Carbon | 8.19 | 0.340 | 6 | 3 | 1 | | |
| Ammonium | 0.0207 | 0.0028 | 10 | 3 | 1 | | |
| Ammonium | 0.103 | 0.0054 | 12 | 3 | 1 | | |
| Nitrate + Nitrite | 0.142 | 0.0071 | 11 | 2 | 1 | | |
| Nitrate + Nitrite | 0.425 | 0.0147 | 13 | 2 | 1 | | |
| Orthophosphate | 0.0230 | 0.0027 | 11 | 2 | 1 | | |
| Orthophosphate | 0.0453 | 0.0023 | 12 | 3 | 1 | | |
| | | | | | | | |
| Spring 2018 | | | | | | | |
| Particulate Carbon | 1.66 | 0.150 | 7 | 3 | | | |
| Particulate Nitrogen | 0.257 | 0.0250 | 7 | 3 | | | |
| Particulate Phosphorus | 0.0281 | 0.0029 | 7 | 3 | | | |
| Total Suspended Solids | 61.4 | 5.94 | 14 | 2 | 1 | | |
| Total Dissolved Nitrogen | 0.311 | 0.1740 | 13 | | | 1 | |
| Total Dissolved Nitrogen | 0.532 | 0.0229 | 8 | 7 | | | |
| Total Dissolved Phosphorus | 0.0140 | 0.0041 | 11 | 2 | 1 | | |
| Total Dissolved Phosphorus | 0.0584 | 0.0046 | 10 | 6 | | | |
| Dissolved Organic Carbon | 4.10 | 0.171 | 9 | 3 | | | |
| Dissolved Organic Carbon | 8.03 | 0.304 | 8 | 4 | | | |
| Ammonium | 0.064 | 0.0035 | 10 | 4 | | | |
| Ammonium | 0.321 | 0.0134 | 13 | 1 | 2 | | |
| Nitrate + Nitrite | 0.0948 | 0.0107 | 14 | | | 1 | |
| Nitrate + Nitrite | 0.713 | 0.0371 | 14 | 2 | | 1 | |
| Orthophosphate | 0.0313 | 0.0029 | 9 | 5 | | | |
| Orthophosphate | 0.520 | 0.0030 | 11 | 4 | 1 | | |

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

| | | | 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 | | |
| Fall 2017 | | | | | | | |
| Total Dissolved Nitrogen | 0.353 | 0.284-0.47 | 9 | 3 | 1 | | |
| Total Dissolved Nitrogen | 0.705 | 0.610-0.742 | 14 | 1 | | | |
| Total Dissolved Phosphorus | 0.0190 | 0.0230- 0.0322* | | 1 | 13 | | |
| Total Dissolved Phosphorus | 0.0190 | 0.0322 | | 2 | 14 | | |
| Ammonium | 0.021 | 0.016-0.028* | 10 | 2 | 2 | | |
| Ammonium | 0.106 | 0.089-0.111 | 15 | 1 | | | |
| Nitrate + Nitrite | 0.140 | 0.128-0.159 | 13 | 1 | | | |
| Nitrate + Nitrite | 0.420 | 0.400-0.456 | 16 | | | | |
| Orthophosphate | 0.0223 | 0.0189-0.030* | 9 | 4 | 1 | | |
| Orthophosphate | 0.0446 | 0.0420-0.0506 | 15 | 1 | | | |
| Dissolved Organic Carbon | 3.00 | 2.58-3.40 | 8 | 2 | | | |
| Dissolved Organic Carbon | 8.00 | 7.80-8.93 | 10 | | | | |
| Total Suspended Solids | 25.0 | 51.0-58.1 | 10 | 4 | 2 | | |
| | | | | | | | |
| Spring 2018 | | | | | | | |
| Total Dissolved Nitrogen | 0.254 | 0.226-0.932 | 10 | 2 | 2 | | |
| Total Dissolved Nitrogen | 0.536 | 0.491-0.571 | 14 | | | | |
| Total Dissolved Phosphorus | 0.0130 | 0.0080- 0.0263* | 6 | 3 | 5 | | |
| Total Dissolved Phosphorus | 0.0560 | 0.0514-0.0658 | 12 | 4 | | | |
| Ammonium | 0.0680 | 0.059-0.071* | 11 | 3 | | | |
| Ammonium | 0.326 | 0.290-0.351 | 15 | 1 | | | |
| Nitrate + Nitrite | 0.0981 | 0.0560-0.104 | 14 | | 1 | | |
| Nitrate + Nitrite | 0.714 | 0.590-0.773 | 16 | 1 | | | |
| Orthophosphate | 0.0312 | 0.0269- 0.0361* | 9 | 5 | | | |
| Orthophosphate | 0.0513 | 0.0449-0.0569 | 14 | 3 | | | |
| Dissolved Organic Carbon | 4.00 | 3.89-4.45 | 11 | 1 | | | |
| Dissolved Organic Carbon | 8.00 | 7.37-8.46 | 12 | | | | |
| Total Suspended Solids | 67.4 | 47.7-66.3 | 11 | 3 | 3 | | |

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

University of Connecticut Center for Environmental Science and Engineering (UCONN)

| | Fall 2017 | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---------------|-----------|---------------|---------------|-------------|
| | Reported | Prepared | Percent | Reported | Prepared | Percent |
| | Concentration | Concentration | Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.3080 | 0.3530 | 87.3 | 0.2680 | 0.2540 | 105.5 |
| TDN (mg N/L) | 0.6780 | 0.7050 | 96.2 | 0.5710 | 0.5360 | 106.5 |
| TDP (mg P/L) | 0.0310 | 0.0190 | 163.2 | 0.0150 | 0.0130 | 115.4 |
| TDP (mg P/L) | 0.0600 | 0.0390 | 153.8 | 0.0550 | 0.0560 | 98.2 |
| NH4 (mg N/L) | 0.0170 | 0.0210 | 81.0 | 0.0690 | 0.0680 | 101.5 |
| NH4 (mg N/L) | 0.1040 | 0.1060 | 98.1 | 0.3300 | 0.3260 | 101.2 |
| NO23 (mg N/L) | 0.1440 | 0.1401 | 102.8 | 0.0990 | 0.0981 | 100.9 |
| NO23 (mg N/L) | 0.4260 | 0.4202 | 101.4 | 0.7220 | 0.7144 | 101.1 |
| PO4 (mg P/L) | 0.0260 | 0.0223 | 116.6 | 0.0280 | 0.0312 | 89.7 |
| PO4 (mg P/L) | 0.0470 | 0.045 | 105.4 | 0.0560 | 0.0513 | 109.2 |
| PC (mg C/L) | 0.9220 | NA NA | NA | 1.7480 | NA | NA |
| PN (mg N/L) | 0.1280 | NA | NA | 0.2500 | NA | NA |
| PP (mg P/L) | 0.0140 | NA | NA | 0.0265 | NA | NA |
| CHL (ug/L) | 7.25 | NA NA | NA | 9.31 | NA | NA |
| DOC (mg C/L) | * | 3.00 | * | 4.08 | 4.00 | 102.0 |
| DOC (mg C/L) | * | 8.00 | * | 7.96 | 8.00 | 99.5 |
| TSS (mg/L) | 22.3 | 25.0 | 89.2 | 64.7 | 67.4 | 96.0 |

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

Academy of Natural Science of Philadelphia (ACNAT)

| | Fall 2017 | | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---|---------------|-----------|---------------|---------------|-------------|
| | Reported | | Prepared | Percent | Reported | Prepared | Percent |
| | Concentration | | Concentration | Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.2840 | | 0.3530 | 80.5 | 0.2570 | 0.2540 | 101.2 |
| TDN (mg N/L) | 0.6100 | W | 0.7050 | 86.5 | 0.5170 | 0.5360 | 96.5 |
| TDP (mg P/L) | 0.0233 | | 0.0190 | 122.6 | 0.0152 | 0.0130 | 116.9 |
| TDP (mg P/L) | 0.0482 | | 0.0390 | 123.6 | 0.0551 | 0.0560 | 98.4 |
| NH4 (mg N/L) | 0.0222 | | 0.0210 | 105.7 | 0.0650 | 0.0680 | 95.6 |
| NH4 (mg N/L) | 0.1030 | | 0.1060 | 97.2 | 0.3170 | 0.3260 | 97.2 |
| NO23 (mg N/L) | 0.1360 | | 0.1401 | 97.1 | 0.0953 | 0.0981 | 97.1 |
| NO23 (mg N/L) | 0.4190 | | 0.4202 | 99.7 | 0.7200 | 0.7144 | 100.8 |
| PO4 (mg P/L) | 0.0257 | | 0.0223 | 115.2 | 0.0329 | 0.0312 | 105.4 |
| PO4 (mg P/L) | 0.0488 | | 0.0446 | 109.4 | 0.0517 | 0.0513 | 100.8 |
| PC (mg C/L) | 0.7900 | W | NA | NA | * | NA | NA |
| PN (mg N/L) | 0.1250 | | NA | NA | * | NA | NA |
| PP (mg P/L) | 0.0271 | W | NA | NA | 0.0319 | NA | NA |
| CHL (ug/L) | 10.19 | | NA | NA | 10.45 | NA | NA |
| DOC (mg C/L) | * | | 3.00 | * | * | 4.00 | * |
| DOC (mg C/L) | * | | 8.00 | * | * | 8.00 | * |
| TSS (mg/L) | 21.6 | | 25.0 | 86.4 | 59.8 | 67.4 | 88.7 |

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

[&]quot;W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

[&]quot;W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Virginia Institute of Marine Science, Analytical Service Center (VIMS)

| | Fall 2017 | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---------------|-----------|---------------|---------------|-------------|
| | Reported | Prepared | Percent | Reported | Prepared | Percent |
| | Concentration | Concentration | Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.3361 | 0.3530 | 95.2 | 0.3353 | 0.2540 | 132.0 |
| TDN (mg N/L) | 0.6580 | 0.7050 | 93.3 | 0.5587 | 0.5360 | 104.2 |
| TDP (mg P/L) | 0.0266 | 0.0190 | 140.0 | 0.0096 | 0.0130 | 73.8 |
| TDP (mg P/L) | 0.0514 | 0.0390 | 131.8 | 0.0530 | 0.0560 | 94.6 |
| NH4 (mg N/L) | 0.0160 | 0.0210 | 76.2 | 0.0614 | 0.0680 | 90.3 |
| NH4 (mg N/L) | 0.0974 | 0.1060 | 91.9 | 0.3118 | 0.3260 | 95.6 |
| NO23 (mg N/L) | 0.1278 | 0.1401 | 91.2 | 0.0950 | 0.0981 | 96.8 |
| NO23 (mg N/L) | 0.4280 | 0.4202 | 101.9 | 0.6946 | 0.7144 | 97.2 |
| PO4 (mg P/L) | 0.0208 | 0.0223 | 93.3 | 0.0269 | 0.0312 | 86.2 |
| PO4 (mg P/L) | 0.0440 | 0.0446 | 98.7 | 0.0480 | 0.0513 | 93.6 |
| PC (mg C/L) | 0.9790 | NA | NA | 1.7705 | NA | NA |
| PN (mg N/L) | 0.1230 | NA | NA | 0.2820 | NA | NA |
| PP (mg P/L) | 0.0187 | NA | NA | 0.0283 | NA | NA |
| CHL (ug/L) | 11.46 | NA | NA | 7.96 | NA | NA |
| DOC (mg C/L) | * | 3.00 | * | * | 4.00 | * |
| DOC (mg C/L) | * | 8.00 | * | * | 8.00 | * |
| TSS (mg/L) | 25.1 | 25.0 | 100.4 | 64.5 | 67.4 | 95.7 |

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

Hampton Roads Sanitation District, Central Environmetal Laboratory (HRSD)

| | Fall 2017 | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---------------|-----------|---------------|---------------|-------------|
| | Reported | Prepared | Percent | Reported | Prepared | Percent |
| | Concentration | Concentration | Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | * | 0.3530 | * | * | 0.2540 | * |
| TDN (mg N/L) | 0.7420 | 0.7050 | 105.2 | 0.4910 | 0.5360 | 91.6 |
| TDP (mg P/L) | * | 0.0190 | * | * | 0.0130 | * |
| TDP (mg P/L) | 0.0550 | 0.0390 | 141.0 | 0.0640 | 0.0560 | 114.3 |
| NH4 (mg N/L) | * | 0.0210 | * | * | 0.0680 | * |
| NH4 (mg N/L) | 0.0990 | 0.1060 | 93.4 | 0.3160 | 0.3260 | 96.9 |
| NO23 (mg N/L) | * | 0.1401 | * | * | 0.0981 | * |
| NO23 (mg N/L) | 0.4390 | 0.4202 | 104.5 | 0.7390 | 0.7144 | 103.4 |
| PO4 (mg P/L) | * | 0.0223 | * | * | 0.0312 | * |
| PO4 (mg P/L) | 0.0430 | 0.0446 | 96.4 | 0.0500 | 0.0513 | 97.5 |
| 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) | 8.83 | NA | NA | 5.30 | NA | NA |
| DOC (mg C/L) | 3.23 | 3.00 | 107.7 | 4.33 | 4.00 | 108.3 |
| DOC (mg C/L) | 8.23 | 8.00 | 102.9 | 8.43 | 8.00 | 105.4 |
| TSS (mg/L) | 24.1 | 25.0 | 96.4 | 65.8 | 67.4 | 97.6 |

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

[&]quot;W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

[&]quot;W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

University of Maryland, Horn Point Laboratory (HPL)

| | Fall 2017 | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---------------|-----------|---------------|---------------|-------------|
| | Reported | Prepared | Percent | Reported | Prepared | Percent |
| | Concentration | Concentration | Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.3430 | 0.3530 | 97.2 | 0.2480 | 0.2540 | 97.6 |
| TDN (mg N/L) | 0.7100 | 0.7050 | 100.7 | 0.5280 | 0.5360 | 98.5 |
| TDP (mg P/L) | 0.0279 | 0.0190 | 146.8 | 0.0143 | 0.0130 | 110.0 |
| TDP (mg P/L) | 0.0571 | 0.0390 | 146.4 | 0.0596 | 0.0560 | 106.4 |
| NH4 (mg N/L) | 0.0201 | 0.0210 | 95.7 | 0.0654 | 0.0680 | 96.2 |
| NH4 (mg N/L) | 0.1080 | 0.1060 | 101.9 | 0.3270 | 0.3260 | 100.3 |
| NO23 (mg N/L) | 0.1390 | 0.1401 | 99.2 | 0.0975 | 0.0981 | 99.4 |
| NO23 (mg N/L) | 0.4120 | 0.4202 | 98.0 | 0.7300 | 0.7144 | 102.2 |
| PO4 (mg P/L) | 0.0214 | 0.0223 | 96.0 | 0.0322 | 0.0312 | 103.2 |
| PO4 (mg P/L) | 0.0439 | 0.0446 | 98.4 | 0.0490 | 0.0513 | 95.5 |
| PC (mg C/L) | 0.9540 | NA | NA | 1.3750 | NA | NA |
| PN (mg N/L) | 0.1340 | NA | NA | 0.2105 | NA | NA |
| PP (mg P/L) | 0.0248 | NA | NA | 0.0340 | NA | NA |
| CHL (ug/L) | 6.67 | NA | NA | 7.66 | NA | NA |
| DOC (mg C/L) | * | 3.00 | * | * | 4.00 | * |
| DOC (mg C/L) | * | 8.00 | * | * | 8.00 | * |
| TSS (mg/L) | 24.8 | 25.0 | 99.2 | 65.2 | 67.4 | 96.7 |

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

Delaware Department of Natural Resources (DNREC)

| | Fall 2017 | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|--------------------|--------------------|---------------|-------------|------------------------------|---------------|-------------|
| | Reported | Prepared | l Percent | Reported | Prepared | Percent |
| | Concentration | Concentration | n Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.3540 | 0.3530 | 100.3 | 0.2530 | 0.2540 | 99.6 |
| TDN (mg N/L) | 0.7040 | 0.7050 | 99.9 | 0.5460 | 0.5360 | 101.9 |
| TDP (mg P/L) | 0.0226 | 0.0190 | 118.9 | 0.0102 | 0.0130 | 78.5 |
| TDP (mg P/L) | 0.0459 | 0.0390 | 117.7 | 0.0627 | 0.0560 | 112.0 |
| NH4 (mg N/L) | 0.0243 | 0.0210 | 115.7 | 0.0643 | 0.0680 | 94.6 |
| NH4 (mg N/L) | 0.1080 | 0.1060 | 101.9 | 0.3230 | 0.3260 | 99.1 |
| NO23 (mg N/L) | 0.1380 | 0.140 | 98.5 | 0.0970 | 0.0981 | 98.9 |
| NO23 (mg N/L) | 0.4170 | 0.4202 | 99.2 | 0.7240 | 0.7144 | 101.3 |
| PO4 (mg P/L) | 0.0210 | 0.0223 | 94.2 | 0.0356 | 0.0312 | 114.1 |
| PO4 (mg P/L) | 0.0440 | 0.0446 | 98.7 | 0.0569 | 0.0513 | 110.9 |
| PC (mg C/L) | 0.9155 | NA NA | NA NA | 1.3900 | NA | NA |
| PN (mg N/L) | 0.1315 | NA NA | NA NA | 0.2130 | NA | NA |
| PP (mg P/L) | 0.0166 | N/A | NA NA | 0.0286 | NA | NA |
| CHL (ug/L) | 12.73 | N/A | NA NA | 9.80 | NA | NA |
| DOC (mg C/L) | 2.97 | 3.00 | 99.00 | 4.05 | 4.00 | 101.2 |
| DOC (mg C/L) | 7.83 | 8.00 | 97.88 | 7.37 | 8.00 | 92.1 |
| TSS (mg/L) | 24.9 | 25.0 | 99.6 | 65.9 | 67.4 | 97.8 |
| * NI= =====I= ==== | t to a subtain and | | | and the second second second | | • |

^{*} 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

[&]quot;W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Division of Consolidated Laboratory Services (DCLS)

| | Fall 2017 | | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---|---------------|-----------|---------------|---------------|-------------|
| | Reported | | Prepared | Percent | Reported | Prepared | Percent |
| | Concentration | | Concentration | Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.3550 | | 0.3530 | 100.5666 | 0.2760 | 0.2540 | 108.7 |
| TDN (mg N/L) | 0.7030 | | 0.7050 | 99.7163 | 0.5380 | 0.5360 | 100.4 |
| TDP (mg P/L) | 0.0230 | | 0.0190 | 121.0526 | 0.0150 | 0.0130 | 115.4 |
| TDP (mg P/L) | 0.0440 | | 0.0390 | 112.8205 | 0.0580 | 0.0560 | 103.6 |
| NH4 (mg N/L) | 0.0280 | W | 0.0210 | 133.3333 | 0.0610 | 0.0680 | 89.7 |
| NH4 (mg N/L) | 0.1110 | | 0.1060 | 104.7170 | 0.3190 | 0.3260 | 97.9 |
| NO23 (mg N/L) | 0.1420 | | 0.1401 | 101.3562 | 0.0980 | 0.0981 | 99.9 |
| NO23 (mg N/L) | 0.4220 | | 0.4202 | 100.4284 | 0.7210 | 0.7144 | 100.9 |
| PO4 (mg P/L) | 0.0220 | | 0.0223 | 98.6547 | 0.0300 | 0.0312 | 96.2 |
| PO4 (mg P/L) | 0.0440 | | 0.0446 | 98.6547 | 0.0530 | 0.0513 | 103.3 |
| PC (mg C/L) | 0.9410 | | NA | NA | 1.7150 | NA | NA |
| PN (mg N/L) | 0.1360 | | NA | NA | 0.2575 | NA | NA |
| PP (mg P/L) | 0.0186 | | NA | NA | 0.0266 | NA | NA |
| CHL (ug/L) | 9.17 | | NA | NA | 7.10 | NA | NA |
| DOC (mg C/L) | 3.02 | | 3.00 | 100.67 | 4.08 | 4.00 | 102.0 |
| DOC (mg C/L) | 7.80 | | 8.00 | 97.50 | 8.20 | 8.00 | 102.5 |
| TSS (mg/L) | 25.0 | | 25.0 | 100.0 | 59.0 | 67.4 | 87.5 |

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

Unversity of Maryland, Chesapeake Biological Laboratory (CBL)

| | Fall 2017 | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---------------|-----------|---------------|---------------|-------------|
| | Reported | Prepared | Percent | Reported | Prepared | Percent |
| | Concentration | Concentration | Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.3600 | 0.3530 | 102.0 | 0.2460 | 0.2540 | 96.9 |
| TDN (mg N/L) | 0.7190 | 0.7050 | 102.0 | 0.5130 | 0.5360 | 95.7 |
| TDP (mg P/L) | 0.0259 | 0.0190 | 136.3 | 0.0123 | 0.0130 | 94.6 |
| TDP (mg P/L) | 0.0515 | 0.0390 | 132.1 | 0.0566 | 0.0560 | 101.1 |
| NH4 (mg N/L) | 0.0210 | 0.0210 | 100.0 | 0.0640 | 0.0680 | 94.1 |
| NH4 (mg N/L) | 0.1080 | 0.1060 | 101.9 | 0.3340 | 0.3260 | 102.5 |
| NO23 (mg N/L) | 0.1430 | 0.1401 | 102.1 | 0.0980 | 0.0981 | 99.9 |
| NO23 (mg N/L) | 0.4160 | 0.4202 | 99.0 | 0.7174 | 0.7144 | 100.4 |
| PO4 (mg P/L) | 0.0218 | 0.0223 | 97.8 | 0.0326 | 0.0312 | 104.5 |
| PO4 (mg P/L) | 0.0451 | 0.0446 | 101.1 | 0.0542 | 0.0513 | 105.7 |
| PC (mg C/L) | 0.9185 | NA | NA | 1.8400 | NA | NA |
| PN (mg N/L) | 0.1290 | NA | NA | 0.2830 | NA | NA |
| PP (mg P/L) | 0.0189 | NA | NA | 0.0289 | NA | NA |
| CHL (ug/L) | 7.91 | NA | NA | 6.41 | NA | NA |
| DOC (mg C/L) | 2.88 | 3.00 | 96.00 | 3.89 | 3.999 | 97.3 |
| DOC (mg C/L) | 7.90 | 8.00 | 98.75 | 7.82 | 7.999 | 97.8 |
| TSS (mg/L) | 23.9 | 25.0 | 95.6 | 65.6 | 67.4 | 97.3 |

^{*} 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

[&]quot;W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Virginia Polytechnic Institute, Occoquan Watershed Monitoring Laboratory (OWML)

| | Fall 2017 | | Fall 2017 | Fall 2017 | Spring 2018 | | Spring 2018 | Spring 2018 |
|---------------|---------------|---|---------------|-----------|---------------|---|---------------|-------------|
| | Reported | | Prepared | Percent | Reported | | Prepared | Percent |
| | Concentration | | Concentration | Recovered | Concentration | l | Concentration | Recovered |
| TDN (mg N/L) | 0.4700 | W | 0.3530 | 133.1 | 0.2490 | | 0.2540 | 98.0 |
| TDN (mg N/L) | 0.6600 | | 0.7050 | 93.6 | 0.5570 | | 0.5360 | 103.9 |
| TDP (mg P/L) | 0.0310 | | 0.0190 | 163.2 | 0.0080 |) | 0.0130 | 61.5 |
| TDP (mg P/L) | 0.0550 | | 0.0390 | 141.0 | 0.0610 |) | 0.0560 | 108.9 |
| NH4 (mg N/L) | 0.0200 | | 0.0210 | 95.2 | 0.0660 | | 0.0680 | 97.1 |
| NH4 (mg N/L) | 0.1100 | | 0.1060 | 103.8 | 0.3510 | W | 0.3260 | 107.7 |
| NO23 (mg N/L) | 0.1590 | W | 0.1401 | 113.5 | 0.0990 |) | 0.0981 | 100.9 |
| NO23 (mg N/L) | 0.4560 | W | 0.4202 | 108.5 | 0.7730 | | 0.7144 | 108.2 |
| PO4 (mg P/L) | 0.0300 | W | 0.0223 | 134.5 | 0.0340 |) | 0.0312 | 109.0 |
| PO4 (mg P/L) | 0.0490 | | 0.0446 | 109.9 | 0.0560 |) | 0.0513 | 109.2 |
| PC (mg C/L) | 0.9170 | | NA | NA | 1.6390 | | NA | NA |
| PN (mg N/L) | 0.1610 | W | NA | NA | 0.2820 |) | NA | NA |
| PP (mg P/L) | * | | NA | NA | * | : | NA | NA |
| CHL (ug/L) | 8.85 | | NA | NA | 9.52 | | NA | NA |
| DOC (mg C/L) | 3.05 | | 3.00 | 101.7 | 4.10 |) | 4.00 | 102.5 |
| DOC (mg C/L) | 8.37 | | 8.00 | 104.6 | 8.00 | | 8.00 | 100.0 |
| TSS (mg/L) | 19.8 | | 25.0 | 79.2 | 64.1 | | 67.4 | 95.1 |

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

Maryland Department of Health (MDH)

| | Fall 2017 | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---------------|-------------|---------------|---------------|-------------|
| | Reported | Prepare | d Percent | Reported | Prepared | Percent |
| | Concentration | Concentration | n Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.3390 | 0.353 | 96.0 | 0.2430 | 0.2540 | 95.7 |
| TDN (mg N/L) | 0.6950 | 0.705 | 98.6 | 0.5080 | 0.5360 | 94.8 |
| TDP (mg P/L) | 0.0265 | 0.019 | 0 139.5 | 0.0134 | 0.0130 | 103.1 |
| TDP (mg P/L) | 0.0518 | 0.039 | 0 132.8 | 0.0514 | 0.0560 | 91.8 |
| NH4 (mg N/L) | 0.0210 | 0.021 | 0 100.0 | 0.0591 | 0.0680 | 86.9 |
| NH4 (mg N/L) | 0.1070 | 0.106 | 0 100.9 | 0.3180 | 0.3260 | 97.5 |
| NO23 (mg N/L) | 0.1480 | 0.140 | 1 105.6 | 0.1040 | 0.0981 | 106.0 |
| NO23 (mg N/L) | 0.4430 | 0.420 | 2 105.4 | 0.7520 | 0.7144 | 105.3 |
| PO4 (mg P/L) | 0.0189 | 0.022 | 3 84.8 | 0.0325 | 0.0312 | 104.2 |
| PO4 (mg P/L) | 0.0420 | 0.044 | 6 94.2 | 0.0528 | 0.0513 | 102.9 |
| PC (mg C/L) | 0.9130 | N | A NA | 1.7560 | NA NA | NA |
| PN (mg N/L) | 0.1350 | N | A NA | 0.2690 | NA | NA |
| PP (mg P/L) | 0.0157 | N | A NA | 0.0230 | NA NA | NA |
| CHL (ug/L) | 9.87 | N | A NA | 7.18 | NA NA | NA |
| DOC (mg C/L) | 3.10 | 3.0 | 0 103.3 | 4.06 | 4.00 | 101.5 |
| DOC (mg C/L) | 8.10 | 8.0 | 0 101.3 | 8.10 | 8.00 | 101.3 |
| TSS (mg/L) | 23.4 | 25. | 0 93.6 | 66.3 | 67.4 | 98.4 |

^{*} 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

[&]quot;W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Massachusetts Water Resource Authority, Central Laboratory (MWRA)

| | Fall 2017 | | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---|---------------|-----------|---------------|---------------|-------------|
| | Reported | | Prepared | Percent | Reported | Prepared | Percent |
| | Concentration | | Concentration | Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.3610 | | 0.3530 | 102.3 | 0.2610 | 0.2540 | 102.8 |
| TDN (mg N/L) | 0.6850 | | 0.7050 | 97.2 | 0.5430 | 0.5360 | 101.3 |
| TDP (mg P/L) | 0.0327 | | 0.0190 | 172.1 | 0.0165 | 0.0130 | 126.9 |
| TDP (mg P/L) | 0.0610 | | 0.0390 | 156.4 | 0.0672 | 0.0560 | 120.0 |
| NH4 (mg N/L) | 0.0201 | | 0.0210 | 95.7 | 0.0615 | 0.0680 | 90.4 |
| NH4 (mg N/L) | 0.1010 | | 0.1060 | 95.3 | 0.3250 | 0.3260 | 99.7 |
| NO23 (mg N/L) | 0.1390 | | 0.1401 | 99.2 | 0.0938 | 0.0981 | 95.6 |
| NO23 (mg N/L) | 0.4500 | | 0.4202 | 107.1 | 0.7050 | 0.7144 | 98.7 |
| PO4 (mg P/L) | 0.0250 | W | 0.0223 | 112.1 | 0.0320 | 0.0312 | 102.6 |
| PO4 (mg P/L) | 0.0506 | | 0.0446 | 113.5 | 0.0534 | 0.0513 | 104.1 |
| PC (mg C/L) | 0.9370 | | NA | NA | 1.7300 | NA | NA |
| PN (mg N/L) | 0.1275 | | NA | NA | 0.2600 | NA | NA |
| PP (mg P/L) | 0.0194 | | NA | NA | 0.0259 | NA | NA |
| CHL (ug/L) | 14.9 | | NA | NA | 9.07 | NA | NA |
| DOC (mg C/L) | * | | 3.00 | * | * | 4.00 | * |
| DOC (mg C/L) | * | | 8.00 | * | * | 8.00 | * |
| TSS (mg/L) | 19.6 | | 25.0 | 78.4 | 50.7 | 67.4 | 75.2 |

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

Old Dominion University, Water Quality Laboratory (ODU)

| | Fall 2017 | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---------------|-----------|---------------|---------------|-------------|
| | Reported | Prepared | Percent | Reported | Prepared | Percent |
| | Concentration | Concentration | Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.3690 | 0.3530 | 104.5 | 0.2860 | 0.2540 | 112.6 |
| TDN (mg N/L) | 0.7290 | 0.7050 | 103.4 | 0.5290 | 0.5360 | 98.7 |
| TDP (mg P/L) | 0.0279 | 0.0190 | 146.8 | 0.0133 | 0.0130 | 102.3 |
| TDP (mg P/L) | 0.0575 | 0.0390 | 147.4 | 0.0576 | 0.0560 | 102.9 |
| NH4 (mg N/L) | 0.0218 | 0.0210 | 103.8 | 0.0595 | 0.0680 | 87.5 |
| NH4 (mg N/L) | 0.1040 | 0.1060 | 98.1 | 0.3210 | 0.3260 | 98.5 |
| NO23 (mg N/L) | 0.1430 | 0.1401 | 102.1 | 0.0971 | 0.0981 | 99.0 |
| NO23 (mg N/L) | 0.4190 | 0.4202 | 99.7 | 0.7130 | 0.7144 | 99.8 |
| PO4 (mg P/L) | 0.0222 | 0.0223 | 99.6 | 0.0304 | 0.0312 | 97.4 |
| PO4 (mg P/L) | 0.0447 | 0.0446 | 100.2 | 0.0508 | 0.0513 | 99.0 |
| PC (mg C/L) | 0.9570 | NA | NA | 1.6350 | NA | NA |
| PN (mg N/L) | 0.1340 | NA | NA | 0.2605 | NA | NA |
| PP (mg P/L) | 0.0179 | NA | NA | 0.0277 | NA | NA |
| CHL (ug/L) | 10.07 | NA | NA | 8.33 | NA | NA |
| DOC (mg C/L) | 2.92 | 3.00 | 97.3 | 3.97 | 4.00 | 99.3 |
| DOC (mg C/L) | 7.97 | 8.00 | 99.6 | 7.89 | 8.00 | 98.6 |
| TSS (mg/L) | 22.1 | 25.0 | 88.4 | 65.7 | 67.4 | 97.5 |
| + NII | | | | | | |

^{*} 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

[&]quot;W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Pennsylvania Department of Environmental Protection, Bureau of Laboratories (PADEP)

| | Fall 2017 | Fall 2017 | Fall 2017 | Spring 2018 | | Spring 2018 | Spring 2018 |
|---------------|---------------|---------------|-----------|---------------|---|---------------|-------------|
| | Reported | Prepared | Percent | Reported | | Prepared | Percent |
| | Concentration | Concentration | Recovered | Concentration | ı | Concentration | Recovered |
| TDN (mg N/L) | * | 0.3530 | * | * | | 0.2540 | * |
| TDN (mg N/L) | 0.7300 | 0.7050 | 103.5 | 0.5300 | | 0.5360 | 98.9 |
| TDP (mg P/L) | * | 0.0190 | * | * | | 0.0130 | * |
| TDP (mg P/L) | 0.0640 | 0.0390 | 164.1 | 0.0580 | | 0.0560 | 103.6 |
| NH4 (mg N/L) | * | 0.0210 | * | * | | 0.0680 | * |
| NH4 (mg N/L) | 0.1000 | 0.1060 | 94.3 | 0.2900 | W | 0.3260 | 89.0 |
| NO23 (mg N/L) | * | 0.1401 | * | * | | 0.0981 | * |
| NO23 (mg N/L) | 0.4000 | 0.4202 | 95.2 | 0.7300 | | 0.7144 | 102.2 |
| PO4 (mg P/L) | * | 0.0223 | * | * | | 0.0312 | * |
| PO4 (mg P/L) | 0.0460 | 0.0446 | 103.1 | 0.0510 | | 0.0513 | 99.4 |
| PC (mg C/L) | * | NA | NA | NA | | NA | NA |
| PN (mg N/L) | * | NA | NA | NA | | NA | NA |
| PP (mg P/L) | * | NA | NA | NA | | NA | NA |
| CHL (ug/L) | 11.90 | NA | NA | NA | | NA | NA |
| DOC (mg C/L) | 3.10 | 3.00 | 103.3 | 3.96 | | 4.00 | 99.0 |
| DOC (mg C/L) | 8.23 | 8.00 | 102.9 | 7.75 | | 8.00 | 96.9 |
| TSS (mg/L) | 21.0 | 25.0 | 84.0 | 52.0 | | 67.4 | 77.2 |

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

New Jersey Department of HeathIth (NJDH)

| | Fall 2017 | | Fall 2017 | Fall 2017 | | Spring 2018 | | Spring 2018 | Spring 2018 |
|---------------|---------------|---|---------------|-----------|---|--------------|---|---------------|-------------|
| | Reported | | Prepared | Percent | | Reported | | Prepared | Percent |
| | Concentration | | Concentration | Recovered | С | oncentration | | Concentration | Recovered |
| TDN (mg N/L) | * | | 0.3530 | * | | * | | 0.2540 | * |
| TDN (mg N/L) | * | | 0.7050 | * | | * | | 0.5360 | * |
| TDP (mg P/L) | 0.0322 | | 0.0190 | 169.5 | | 0.0139 | | 0.0130 | 106.9 |
| TDP (mg P/L) | 0.0631 | | 0.0390 | 161.8 | | 0.0524 | | 0.0560 | 93.6 |
| NH4 (mg N/L) | 0.0188 | | 0.0210 | 89.5 | | 0.0666 | | 0.0680 | 97.9 |
| NH4 (mg N/L) | 0.1020 | | 0.1060 | 96.2 | | 0.3140 | | 0.3260 | 96.3 |
| NO23 (mg N/L) | 0.1500 | | 0.1401 | 107.1 | | 0.1010 | | 0.0981 | 103.0 |
| NO23 (mg N/L) | 0.4300 | | 0.4202 | 102.3 | | 0.7140 | | 0.7144 | 99.9 |
| PO4 (mg P/L) | 0.0212 | | 0.0223 | 95.1 | | 0.0270 | | 0.0312 | 86.5 |
| PO4 (mg P/L) | 0.0440 | | 0.0446 | 98.7 | | 0.0449 | W | 0.0513 | 87.5 |
| 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) | 3.40 | W | 3.00 | 113.3 | | 4.45 | | 4.00 | 111.3 |
| DOC (mg C/L) | 8.58 | | 8.00 | 107.3 | | 8.46 | | 8.00 | 105.8 |
| TSS (mg/L) | 23.7 | | 25.0 | 94.8 | | 64.1 | | 67.4 | 95.1 |

^{*} 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

[&]quot;W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

Sprague River Water Quality Laboratory (SRWQL)

| | Fall 2017 | | Fall 2017 | Fall 2017 | Spring 2018 | Spring 2018 | Spring 2018 |
|---------------|---------------|---|---------------|-----------|---------------|---------------|-------------|
| | Reported | | Prepared | Percent | Reported | Prepared | Percent |
| | Concentration | | Concentration | Recovered | Concentration | Concentration | Recovered |
| TDN (mg N/L) | 0.3410 | | 0.3530 | 96.6 | 0.2260 | 0.2540 | 89.0 |
| TDN (mg N/L) | 0.6940 | | 0.7050 | 98.4 | 0.4990 | 0.5360 | 93.1 |
| TDP (mg P/L) | 0.0230 | W | 0.0190 | 121.1 | 0.0130 | 0.0130 | 100.0 |
| TDP (mg P/L) | 0.0740 | | 0.0390 | 189.7 | 0.0570 | 0.0560 | 101.8 |
| NH4 (mg N/L) | 0.0190 | | 0.0210 | 90.5 | 0.0590 | 0.0680 | 86.8 |
| NH4 (mg N/L) | 0.0890 | W | 0.1060 | 84.0 | 0.3020 | 0.3260 | 92.6 |
| NO23 (mg N/L) | 0.1360 | | 0.1401 | 97.1 | 0.0960 | 0.0981 | 97.9 |
| NO23 (mg N/L) | 0.4140 | | 0.4202 | 98.5 | 0.6810 | 0.7144 | 95.3 |
| PO4 (mg P/L) | 0.0230 | | 0.0223 | 103.1 | 0.0300 | 0.0312 | 96.2 |
| PO4 (mg P/L) | 0.0440 | | 0.0446 | 98.7 | 0.0520 | 0.0513 | 101.4 |
| 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) | 7.12 | | NA | NA | 7.89 | NA | NA |
| DOC (mg C/L) | * | | 3.00 | * | * | 4.00 | * |
| DOC (mg C/L) | * | | 8.00 | * | * | 8.00 | * |
| TSS (mg/L) | 25.0 | | 25.0 | 100.0 | 66.3 | 67.4 | 98.4 |

^{*} 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

Microbac (MICRO)

| | Fall 2017 | Fall 2017 | Fall 2017 | Spring 2018 | | Spring 2018 | Spring 2018 |
|---------------|---------------|---------------|-----------|---------------|---|---------------|-------------|
| | Reported | Prepared | Percent | Reported | | Prepared | Percent |
| | Concentration | Concentration | Recovered | Concentration | | Concentration | Recovered |
| TDN (mg N/L) | * | 0.3530 | * | 0.9320 | F | 0.2540 | 366.9 |
| TDN (mg N/L) | * | 0.7050 | * | L | | 0.5360 | NA |
| TDP (mg P/L) | * | 0.0190 | * | L | | 0.0130 | NA |
| TDP (mg P/L) | * | 0.0390 | * | L | | 0.0560 | NA |
| NH4 (mg N/L) | * | 0.0210 | * | * | | 0.0680 | * |
| NH4 (mg N/L) | * | 0.1060 | * | * | | 0.3260 | * |
| NO23 (mg N/L) | * | 0.1401 | * | 0.0560 | F | 0.0981 | 57.1 |
| NO23 (mg N/L) | * | 0.4202 | * | 0.5900 | F | 0.7144 | 82.6 |
| PO4 (mg P/L) | * | 0.0223 | * | L | | 0.0312 | NA |
| PO4 (mg P/L) | * | 0.0446 | * | L | | 0.0513 | NA |
| 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 | 3.20 | | NA | NA |
| DOC (mg C/L) | * | 3.00 | * | 3.94 | | 4.00 | 98.5 |
| DOC (mg C/L) | * | 8.00 | * | 7.97 | | 8.00 | 99.6 |
| TSS (mg/L) | * | 25.0 | * | 57.0 | | 67.4 | 84.6 |

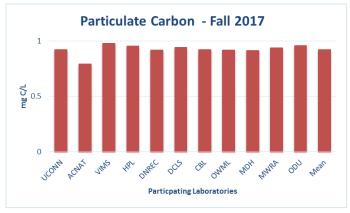
^{*} No sample sent to participant - sample not requested, parameter or concentration range not routine "W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations

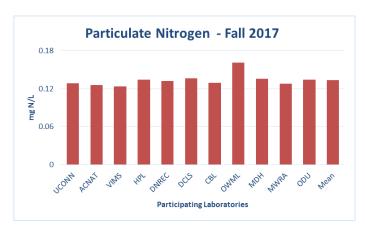
Appendix 1 Cont'. Fall 2017 and Spring 2018 Reported Concentrations, Prepared Concentrations and Recoveries

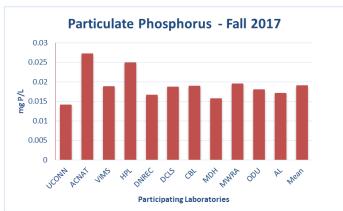
University of Maryland Appalachian Laboratory (AL)

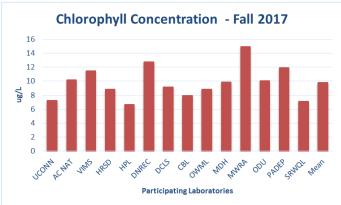
| | Fall 2017 Reported | | Fall 2017 Prepared | Fall 2017 Percent | Spring 2018 Reported | | Spring 2018 Prepared | Spring 2018 Percent |
|---------------|-----------------------|---|-----------------------|----------------------|-------------------------|------------------|-------------------------|------------------------|
| | Concentration | | Concentration | Recovered | Concentration | | Concentration | Recovered |
| TDN (mg N/L) | 0.4019 | | 0.3530 | 113.9 | 0.2728 | | 0.2540 | 107.4 |
| TDN (mg N/L) | 0.7320 | | 0.7050 | 103.8 | 0.5584 | | 0.5360 | 104.2 |
| TDP (mg P/L) | 0.0254 | | 0.0190 | 133.7 | 0.0263 | W | 0.0130 | 202.3 |
| TDP (mg P/L) | 0.0519 | | 0.0390 | 133.1 | 0.0658 | | 0.0560 | 117.5 |
| NH4 (mg N/L) | 0.0204 | | 0.0210 | 97.1 | 0.0708 | | 0.0680 | 104.1 |
| NH4 (mg N/L) | 0.1043 | | 0.1060 | 98.4 | 0.3342 | | 0.3260 | 102.5 |
| NO23 (mg N/L) | 0.1382 | | 0.1401 | 98.6 | 0.0950 | | 0.0981 | 96.8 |
| NO23 (mg N/L) | 0.4121 | | 0.4202 | 98.1 | 0.6999 | | 0.7144 | 98.0 |
| PO4 (mg P/L) | 0.0232 | | 0.0223 | 104.0 | 0.0361 | | 0.0312 | 115.7 |
| PO4 (mg P/L) | 0.0447 | | 0.0446 | 100.2 | 0.0525 | | 0.0513 | 102.3 |
| PC (mg C/L) | * | | NA | NA | * | | NA | NA |
| PN (mg N/L) | * | | NA | NA | * | | NA | NA |
| PP (mg P/L) | 0.0170 | | NA | NA | * | | NA | NA |
| CHL (ug/L) | * | | NA | NA | * | | NA | NA |
| DOC (mg C/L) | 3.21 | | 3.00 | 107.1 | 4.35 | | 4.00 | 108.9 |
| DOC (mg C/L) | 8.93 | W | 8.00 | 111.6 | 8.41 | , and the second | 8.00 | 105.1 |
| TSS (mg/L) | 23.5 | | 25.0 | 94.0 | 47.4 | W | 67.4 | 70.3 |

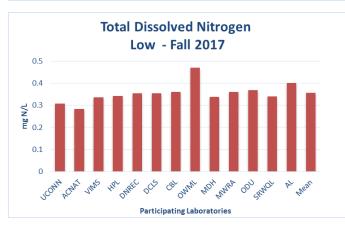
^{*} No sample sent to participant - sample not requested, parameter or concentration range not routine
"W" Warn and "F" Fail based on standard deviation of all participants' reported concentrations











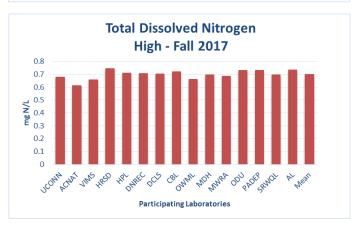
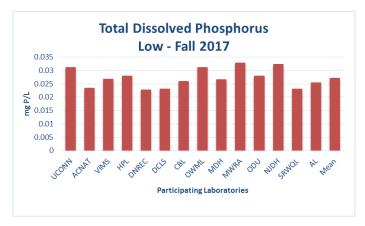
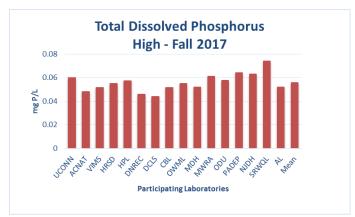
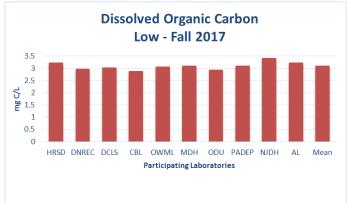
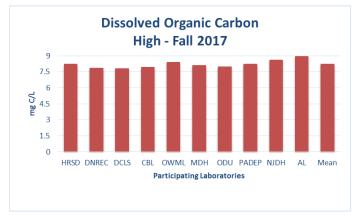


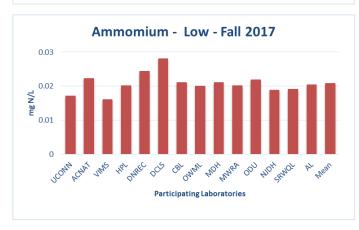
Figure 1. Particulate carbon, nitrogen and phosphorus; chlorophyll *a*, and total dissolved nitrogen. Fall 2017











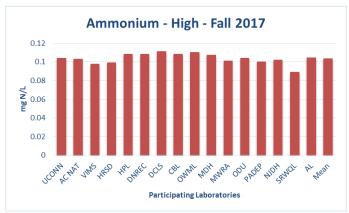
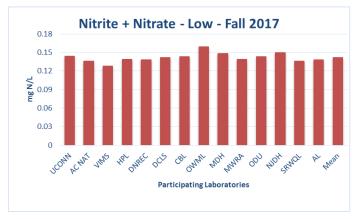
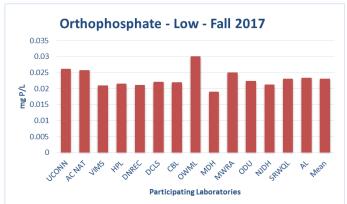


Figure 2. Total dissolved phosphorus; dissolved organic carbon, amd ammonium. Fall 2017







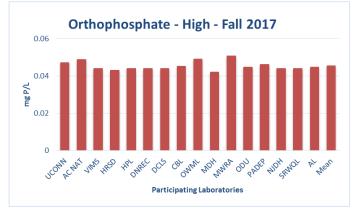




Figure 3. Nitrite plus nitrate, orthophosphate, and total suspended solids. Fall 2017

Participating Laboratories



Figure 4. Particulate carbon, nitrogen and phosphorus; chlorophyll *a*, and total dissolved nitrogen. Spring 2018.

Participating Laboratories



Figure 5. Total dissolved phosphorus; dissolved organic carbon, amd ammonium. Spring 2018.



Figure 6. Nitrite plus nitrate, orthophosphate, and total suspended solids. Spring 2018.