

**North Carolina National Estuarine Research Reserve (NOC)
NERR Nutrient Metadata Report
January– December 2006
Latest Update: May 19, 2025**

I. Data Set and Research Descriptors

1. Principal investigator(s) and contact persons

a) Reserve contacts

Dr. John Fear, Research Coordinator
400 Commerce Ave.
Morehead City, NC 28557
Phone: (252) 808-2808 ext. 224
Fax: (252) 247-3330
Email: john.fear@ncmail.net

Paula Murray, Research Associate
5600 Marvin K. Moss Lane
Wilmington, NC 28409
Phone: (910) 962-2334
Fax: (910) 962-2410
Email: murrayp@uncw.edu

Heather Wells, Research Associate
5600 Marvin K. Moss Lane
Wilmington, NC 28409
Phone: (910) 962-2335
Fax: (910) 962-2410
Email: wellsh@uncw.edu

b) Laboratory contact

Carol Pollard
Email: pollard@vims.edu
VIMS Analytical Service Center
Route 1208
Greate Road
Gloucester Point, VA 23062
(804) 684-7213

2. Research objectives

The objective of this research is to provide baseline information on inorganic nutrient and Chla water quality status within the North Carolina (NOC) NERR. Water samples are

collected, processed, and analyzed for the following parameters: ammonium (NH_4^+), nitrate (NO_3^-), nitrite (NO_2^-), ortho-phosphate, and chlorophyll *a* concentrations. Data collected will assist in understanding the impacts of anthropogenic impacts on the watersheds.

Monthly grab

Monthly grab samples are collected to quantify the spatial and temporal variability of selected nutrients and chlorophyll *a* concentrations in the water column at sites representative of the local estuarine system.

Diel sampling program

Once per month, samples are collected at Research Creek every two hours and eleven minutes through a complete tidal cycle to quantify the short-term temporal variability of selected nutrients and chlorophyll *a* in the water column.

3. Research methods

Monthly grab sampling program:

Monthly grab samples were taken at four stations within the Masonboro Island and Zeke's Island components of the NOCNERR. Samples are taken at each of the principal NOCNERR datasonde stations, which are Research Creek, Loosin Creek, Zeke's Basin and East Cribbings. Efforts are made to collect all samples within a 24 hour time period during an ebb tide after an antecedent dry period of 72 hours. No distinctions are made between neap and spring tide conditions. Replicate ($N=2$) samples are collected by hand at an approximate depth of 10 cm. All samples are collected in amber, wide-mouth, Nalgene sample bottles that are previously acid washed (10% HCl), rinsed (3x) with distilled-deionized water, dried and followed by rinsing (3x) of ambient water prior to collection of the sample. Samples are immediately placed on ice, in a dark cooler and returned to the laboratory.

Once in the NOCNERR laboratory, samples are shaken and processed for chlorophyll *a* and nutrient analyses. Sample processing includes the filtration of samples, then either shipment to the Virginia Institute for Marine Science (VIMS) or in-house analysis. Beginning July 2006, chlorophyll *a* analyses were performed by NOCNERR staff rather than the VIMS laboratory and analysis for phaeophytin was discontinued. For chlorophyll *a* and phaeophytin, volumes of 100 ml are filtered through a 47 mm Whatman GF/F glass microfiber filter (pore size $0.7\ \mu\text{m}$, VIMS method) or a 25mm Millipore glass microfiber filter (chlorophyll *a* only, in-house method) using a vacuum pump and filtering flask. Filters are folded in half, placed in aluminum foil, and stored in a freezer with a desiccant until time of sample shipment or analysis. For nutrient processing, samples are filtered through a 47mm Millipore MF membrane filter (mixed cellulose ester, pore size $0.45\ \mu\text{m}$). The membrane filter is discarded and the liquid filtrate poured into a 125 ml acid washed Nalgene bottle and placed in the freezer until

shipment time. Sample collection bottles are acid washed (10% HCl) and rinsed (3x) using distilled-deionized water between sampling events to avoid any contamination.

Diel sampling program:

Diel samples are taken at the Research Creek datasonde station, located in the Masonboro Island reserve. Samples are collected over a 24 hour time period at intervals of two hours and eleven minutes using an ISCO 6700 auto-sampler. Samples are collected at a fixed depth (0.5m) from the bottom, representing the water mass sampled by the datasonde. No distinction is made between neap and spring tide conditions. Efforts are made to allow for an antecedent dry period of 72 hours prior to sampling. Sampling events are staggered approximately 30 days apart to the best of the research staff's ability. All samples are collected in 1000 ml polyethylene bottles that are acid washed (10% HCL), rinsed (3x) with distilled-deionized water and dried. Samples are stored inside the ISCO sampler and kept cool with ice. Once in the laboratory, samples are shaken and processed for nutrient and chlorophyll *a*, in the same manner as described in previous section; "monthly grab sampling program".

4. Site location and character

The components of North Carolina's National Estuarine Research Reserve (from north to south) are: Currituck Banks, Rachel Carson, Masonboro Island, and Zeke's Island. They are located along the southeast Atlantic coast of the United States. Currently, only data from Masonboro Island and Zeke's Island components are transferred to the CDMO. The four monitoring sites are:

A. Research Creek, Masonboro Island

The first Masonboro Island site (formerly called Masonboro Island (MS)) is 0.72 km north east from the mouth of Whiskey Creek, and east of the Intracoastal Waterway (ICW), in a small navigable channel called Research Creek at 34°09'21.7" latitude and 77° 50'59.9" longitude. The site typically has a salinity range of 20-35 ppt and a tidal range that averages around 1.2 meters. The sole source of freshwater is rain and salinity values as little as 10 ppt have been recorded during periods of heavy rain. The creek bottom is characterized by sand and detritus based sediment with areas of soft mud with a depth ranging from 0.2 to 2.6 m. *Spartina* spp. marsh and dunes surround the site, which is relatively unimpacted by manmade perturbations and it is not accessible to road traffic. The site does experience minimal boat traffic.

B. Loosin Creek, Masonboro Island

The second Masonboro Island site (added in 2002) is 1.2 km east of the ICW, and 2.5 km south west of Masonboro Inlet, in a small navigable channel called Loosin Creek at 34° 10'20.0" latitude and 77° 49'58.1" longitude. The site generally has a salinity range of 22-35 ppt and a tidal range that averages 1.2 meters. The sole source of freshwater is rain and salinity values as little as 15 ppt have been recorded during periods of heavy rain. The creek bottom is characterized by sand and detritus based sediment with areas of soft mud with a depth ranging from 0.1 to 2.5 m. *Spartina* spp. marsh and

dunes surround the site, which is relatively unimpacted by manmade perturbations and it is not accessible to road traffic. The site does experience minimal boat traffic.

C. East Cribbings, Zeke's Island

The first Zeke's Island site (formerly called Zeke's Island (ZI)) is located 1.8 km south of Federal Point boat launch in a tidal basin estuary at 33° 56'23.5" latitude and 77° 56'28.1" longitude. This site receives minimal freshwater input from leakage of the Cape Fear River through the 5.6 km rock jetty that separates the two bodies of water. The site typically has a salinity range of 15-33 ppt, although values as little as 10 ppt have been recorded. Tidal range averages 1.2 meters. Depth varies, but usually can be found to range from 0.5 to 2.7 meters. Bottom type substratum consists of large rocks (the cribbings) with sand and detritus based sediment. There are no pollutants from land. Marsh and dunes surround the site. It is not accessible to road traffic but experiences minimal boat traffic.

D. Zeke's Basin, Zeke's Island

The second Zeke's Island site (added in 2002) is located 0.8 km south east of the Federal Point boat launch in a tidal basin estuary at 33° 57'17.0" latitude and 77° 56'6.0" longitude. This site receives minimal freshwater input from leakage of the Cape Fear River through the 5.6 km rock jetty that separates the two bodies of water. The site has a characteristic salinity range of 12-30 ppt, but values below 10 ppt have been observed and are often associated with periods of heavy rainfall. Tidal range averages 1.2 meters. Depth varies, but typically it can be found to range from 0.1 to 1.8 meters. Bottom type substratum consists of sand and detritus based sediment with a layer of soft sulfuric mud. There are no pollutants from land. Marsh and dunes surround the site. It is not accessible to road traffic but experiences minimal boat traffic.

5. Code variable definitions

Station codes:

nocrcnut – North Carolina Reserve Research Creek Nutrient data

nocecnut - North Carolina Reserve East Cribbing Nutrient data

noclcnut - North Carolina Reserve Loosin Creek Nutrient data

noczbnut - North Carolina Reserve Zeke's Basin Nutrient data

Program Codes: Grab Sampling-1; Diel Sampling-2

See Section 14, Table 2 for comment codes

6. Data collection period

Diel Sampling (All times in EST):

<u>Site</u>	<u>Start Date</u>	<u>Start Time</u>	<u>End Date</u>	<u>End Time</u>
RC	01/12/06	12:30	01/13/06	12:31
RC	02/14/06	13:50	02/15/06	13:51

RC	03/15/06	12:15	03/16/06	07:54
RC	04/10/06	11:30	04/11/06	11:31
RC	05/16/06	14:40	05/17/06	14:41
RC	06/18/06	17:20	06/19/06	17:21
RC	07/10/06	13:05	07/11/06	13:06
RC	08/16/06	08:45	08/17/06	08:46
RC	09/11/06	15:00	09/12/06	15:01
RC	10/10/06	14:55	10/11/06	14:56
RC	11/09/06	14:55	11/10/06	14:56
RC	12/05/06	14:40	12/06/06	14:41

Grab Sampling (All times in EST):

Site	Start Date	Rep 1 Time	Start Date	Rep 2 Time
LC	01/12/06	12:54	01/12/06	12:55
EC	01/12/06	09:18	01/12/06	09:19
ZB	01/12/06	09:51	01/12/06	09:52
RC	01/12/06	12:11	01/12/06	12:12
LC	02/15/06	14:39	02/15/06	14:40
EC	02/15/06	11:10	02/15/06	11:11
ZB	02/15/06	11:39	02/15/06	11:40
RC	02/15/06	14:11	02/15/06	14:12
LC	03/16/06	12:27	03/16/06	12:28
EC	03/16/06	09:58	03/16/06	09:59
ZB	03/16/06	10:32	03/16/06	10:33
RC	03/16/06	13:09	03/16/06	13:10
LC	04/11/06	11:03	04/11/06	11:04
EC	04/11/06	07:57	04/11/06	07:58
ZB	04/11/06	08:23	04/11/06	08:24
RC	04/11/06	11:39	04/11/06	11:40
LC	05/16/06	13:57	05/16/06	13:58
EC	05/16/06	11:56	05/16/06	11:57
ZB	05/16/06	12:17	05/16/06	12:18
RC	05/16/06	14:19	05/16/06	14:20
LC	06/18/06	17:38	06/18/06	17:39
EC	06/18/06	15:32	06/18/06	15:33
ZB	06/18/06	15:43	06/18/06	15:44
RC	06/18/06	16:58	06/18/06	16:59
LC	07/11/06	13:10	07/11/06	13:11
EC	07/11/06	09:59	07/11/06	10:00
ZB	07/11/06	09:35	07/11/06	09:36
RC	07/11/06	13:44	07/11/06	13:45
LC	08/21/06	11:30	08/21/06	11:31
EC	08/21/06	08:51	08/21/06	08:52
ZB	08/21/06	09:26	08/21/06	09:27
RC	08/21/06	10:51	08/21/06	10:52

LC	09/11/06	15:18	09/11/06	15:19
EC	09/11/06	12:40	09/11/06	12:41
ZB	09/11/06	13:05	09/11/06	13:06
RC	09/11/06	14:37	09/11/06	14:38
LC	10/10/06	15:11	10/10/06	15:12
EC	10/10/06	12:29	10/10/06	12:30
ZB	10/10/06	12:51	10/10/06	12:52
RC	10/10/06	14:23	10/10/06	14:24
LC	11/09/06	15:14	11/09/06	15:15
EC	11/09/06	12:45	11/09/06	12:46
ZB	11/09/06	13:05	11/09/06	13:06
RC	11/09/06	14:36	11/09/06	14:37
LC	12/05/06	13:42	12/05/06	13:43
EC	12/05/06	10:09	12/05/06	10:10
ZB	12/05/06	10:32	12/05/06	10:33
RC	12/05/06	14:18	12/05/06	14:19

7. Associated researchers and projects

This effort is part of a larger estuarine observing program including water quality and meteorological monitoring. Additional projects are ongoing and continually changing. Check with the Research Coordinator or other contact person for an updated list of research (see section I.1.).

8. Distribution

National Oceanic and Atmospheric Administration (NOAA)/Estuarine Reserves Division (ERD) retains the right to analyze, synthesize and publish summaries of the NERRS SWMP data. The PI retains the right to be fully credited for having collected and processed the data. Following academic courtesy standards, the PI and NERR site where the data were collected will be contacted and fully acknowledged in any subsequent publications in which any part of the data are used. Manuscripts resulting from the NOAA/OCRM supported research that are produced for publication in open literature, including refereed scientific journals, will acknowledge that the research was conducted under an award from the ERD, OCRM, National Ocean Service (NOS), NOAA. The data set enclosed within the package/transmission is only as good as the quality assurance/quality control procedures outlined by the enclosed metadata reporting statement. The user bears all responsibility for its subsequent use/misuse in any further analyses or comparisons. The Federal government does not assume liability to the Recipient of third persons, nor will the Federal government reimburse or indemnify the Recipient for its liability due to any losses resulting in any way from the use of this data.

NERR water quality, nutrient, and weather data and metadata can be obtained from the Research Coordinator at the individual NERR site (see section I. 1. Principal investigators and contact persons), from the Data Manager at the Centralized Data Management Office (see personnel directory under the general information link on the

CDMO home page) and online at the CDMO home page <http://cdmo.baruch.sc.edu/>. Data are available in text tab-delimited format, Microsoft Excel spreadsheet format and comma-delimited format.

II. Physical Structure Descriptors

9. Entry verification

Monthly nutrient and chlorophyll *a* data files, in excel format, are sent to NOCNERR by the VIMS Analytical Service Center (in July 2006 NOCNERR began analyzing samples for chlorophyll *a* in-house). Files consist of sampling station ID, date and parameter values expressed in unit concentrations. The laboratory supervisor verifies all parameter values in the excel file through cross comparison with laboratory data sheets. The data are copied into an Excel template created by the CDMO and used to generate calculated parameter values. The CDMO nutrient rounding macro rounds all values to the appropriate number of decimal places. All data are visually QA/QC checked by NOCNERR staff. The pre-processed file is then ready to be copied into the EQWin nutrient.eqi file where the data are QA/QC checked and archived in a database. EQWin queries, reports and graphs are used to discover data set anomalies. EQWin is also used to generate statistics, view graphs, create customized queries and reports of the data, cross query the water, weather and nutrient data and finally export the data to CDMO. Heather Wells and Paula Murray were responsible for data management during 2006.

10. Parameter titles and variable names by data category

Data Category	Parameter	Variable Name	Units of Measure
Phosphorus:	Orthophosphate	PO4F	mg/L as P
Nitrogen:	Nitrite + Nitrate, Filtered	NO23F	mg/L as N
	Nitrite, Filtered	NO2F	mg/L as N
	Nitrate, Filtered	NO3F	mg/L as N
	Ammonium, Filtered	NH4F	mg/L as N
	Dissolved Inorganic Nitrogen	DIN	mg/L as N
Other Lab Parameters:			
	Chlorophyll <i>a</i>	CHLA_N	µg/L
	Phaeophytin	PHEA	µg/L

11. Measured and calculated laboratory parameters

a) Parameters measured directly

Nitrogen species:	NO2F, NO23F, NH4F
Phosphorus species:	PO4F

Other: CHLA_N, PHEA

b) Calculated parameters

Nitrogen Species: NO3: NO23F-NO2F
DIN: NO23F+NH4F

12. Limits of detection

Method Detection Limits (MDL), the lowest concentration of a parameter that an analytical procedure can reliably detect, have been established by the VIMS Nutrient Analytical Laboratory. The MDL is determined as 3 times the standard deviation of a minimum of 7 replicates of a single low concentration sample. Table 1 presents the current MDLs; these values are reviewed and revised periodically.

Table 1. Method Detection Limits (MDL) for measured water quality parameters.

Parameter	Variable	MDL	Dates in use
Ammonium	NH4F	0.0054 mg/L as N	2006
Nitrite	NO2F	0.0002 mg/L as N	2006
Nitrate + Nitrite	NO23F	0.0010 mg/L as N	2006
Orthophosphate	PO4F	0.0015 mg/L as P	2006
Chlorophyll	CHLA_N	0.5 µg/L	2006
Phaeophytin	PHEA	0.5 µg/L	2006

13. Laboratory methods

a) Parameter: Ammonia (NH4F)

Method References: Virginia Institute of Marine Science Analytical Service Center. U.S. EPA. 1974. Methods for Chemical Analysis of Water and Wastes, pp. 168-174.

Standard Methods for the Examination of Water and Wastewater, 14th edition. p 410. Method 418A and 418B (1975). Annual Book of ASTM Standards, Part 31. "Water", Standard 1426-74, Method A, p 237 (1976). EPA 600/R-97/072 Method 349.0. Determination of Ammonia in Estuarine and Coastal Waters by Gas Segmented Continuous Flow Colorimetric Analysis. IN: Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Matrices - 2nd Edition. National Exposure Research Laboratory, Office of Research and Development U.S. EPA, Cincinnati, Ohio 45268.

Method Descriptor: Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside. Reaction is heat catalyzed at 37°C and is measured colorimetrically at 660 nm. The range is 0.01 - 2.0 mg/L.

Preservation Method: A 100 ml volume of sample is filtered through a Whatman glass fiber filter (0.45 µm pore size, 47 mm diameter) using a vacuum pump. The pooled filtrate is then filtered through a Millipore membrane filter (0.45 µm pore size, 47 mm diameter). The liquid volume is then poured into a Nalgene bottle and stored at -20°C until sent for analysis.

b) Parameter: Nitrate + Nitrite (NO₂3F), Nitrate (NO₃F), and Nitrite (NO₂F)

Method References: Virginia Institute of Marine Science Analytical Service Center.

SKALAR Method: Nitrate + Nitrite/ Total Dissolved Nitrogen Catnr. 461-353.2 issue 120293/MH/93128060. SKALAR Method 467. U.S. EPA. 1974 Methods for Chemical Analysis of Water and Wastes, pp. 207 -212. Wood, E.D., F.A.G. Armstrong and F.A. Richards. 1967. Determination of nitrate in seawater by cadmium-copper reduction to nitrite. J. Mar. Biol. Assoc. U.K. 47: 23. Grasshoff, K., M. Ehrhardt and K. Kremling. 1983. Methods of Seawater Analysis. Verlag Chemie, Federal Republic of Germany. 419 pp. EPA 600/R-97/072 Method 353.4 Determination of Nitrate and Nitrite in Estuarine and Coastal Waters by Gas Segmented Flow Colorimetric Analysis. IN: Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Matrices - 2nd Edition. National Exposure Research Laboratory, Office of Research and Development U.S. EPA, Cincinnati, Ohio 45268.

Method Descriptor: Nitrate is reduced to nitrite by a copper/cadmium reductor column. The nitrite ion then reacts with sulfanilimide to form a diazo compound. This compound then couples with n-1-naphthylenediamine dihydrochloride to form a reddish/purple azo dye and is read colorimetrically at 540 nm. Nitrate concentration is obtained by subtracting the corresponding nitrite value from the NO₃⁻ + NO₂⁻ concentration. The color development chemistry is the same as that used in Nitrite. Range is 0 -1.2 mg/L.

Preservation Method: A 100 ml volume of sample is filtered through a Whatman glass fiber filter (0.45 µm pore size, 47 mm diameter) using a vacuum pump. The pooled filtrate is then filtered through a Millipore membrane filter (0.45 µm pore size, 47 mm diameter). The liquid volume is then poured into a Nalgene bottle and stored at -20°C until sent for analysis.

c) Parameter: Orthophosphate (PO₄)

Method References: Virginia Institute of Marine Science Analytical Service Center.

SKALAR Method: O-Phosphate / Total Phosphate Catnr. 503-365.1, issue 042993/MH/93-Demo1. Murphy, J. and J.P. Riley. 1962. A modified single solution method for the determination of phosphate in natural waters. Analytica

Chim. Acta 27 : 31-36. EPA 600/R-97/072 Method 365.5 Determination of Orthophosphate in Estuarine and Coastal Waters by Automated Colorimetric Analysis. IN: Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Matrices - 2nd Edition. National Exposure Research Laboratory, Office of Research and Development. U.S. EPA, Cincinnati, Ohio 45268.

Method Descriptor: Ammonium molybdate and antimony potassium tartrate react in a sulfuric acid environment to form an antimony-phospho-molybdo complex, which is reduced to a blue colored complex by ascorbic acid. Reaction is heat catalyzed at 40°C and measured colorimetrically at 880nm. The range is 1-50 ppb.

Preservation Method: A 100 ml volume of sample is filtered through a Whatman glass fiber filter (0.45 µm pore size, 47 mm diameter) using a vacuum pump. The pooled filtrate is then filtered through a Millipore membrane filter (0.45 µm pore size, 47 mm diameter). The liquid volume is then poured into a Nalgene bottle and stored at -20°C until sent for analysis.

d) Parameter: Chlorophyll *a* (CHLA_N), Phaeophytin (PHEA)

Method References: Virginia Institute of Marine Science Analytical Service Center.

Strickland, J.D.H., and Parson, T.R. 1972. A Practical Handbook of Seawater Analysis. Fish. Res. Bd. Canada 167:310.

TD-700 Laboratory Fluorometer Operating Manual. Version 1.8. July 7, 1999. Turner Designs, 845 West Maude Avenue, Sunnyvale, CA 94086.

EPA /600/ R-97/072 - Method 445.0. *In Vitro* Determination of Chlorophyll *a* and Pheophytin *a* in Marine and Freshwater Algae by Fluorescence. Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Matrices Revision 1.2. September 1997.

Using the Turner Designs Model 10 Analog, The 10AU Digital, Or the TD-700 Fluorometer with EPA Method 445.0. January 19, 1999. Turner Designs, 845 West Maude Avenue, Sunnyvale, CA 94086.

Method Descriptor: The two methods for determining chlorophyll *a* given here are with 1) a scanning spectrophotometer and 2) a Turner Design fluorometer. The method used requires filtering a known quantity of water through a glass fiber filter (4.7 cm GF/F). This filter is later ground with a tissue grinder made of teflon/glass. Approximately 1-3mLs of 90% acetone are added to the filter before grinding. Acetone is also used to wash the filter into 17 x 150 test tube with tight fitting cap. The sample is steeped at least 2 hours and not exceeding 24 hours at 4°C, in the dark. The samples are centrifuged and read on a spectrophotometer or fluorometer. If the samples can not be read within that time period, storage in the freezer at -20°C for a few days is acceptable. If

phaeophytin measurements are desired, the sample is acidified and read again.

Preservation Method: A 100 ml volume of sample is filtered through a Whatman glass fiber filter (0.45 µm pore size, 47 mm diameter) using a vacuum pump. The filter is folded in half, wrapped in aluminum foil, and stored with desiccant –20°C until it is sent for analysis.

14. Reporting of missing data, data with concentrations lower than method detection limits, and other comment codes

Nutrient/Chla comment codes and definitions are provided in the following table. Missing data are denoted by a blank cell “ ” and commented coded with an “M”. Laboratories in the NERRS System submit data that are censored at a lower detection rate limit, called the Method Detection Limit or MDL. MDL’s for specific parameters are listed in the Laboratory Methods and Detection Limits Section (Section II, Part 14) of this document. Measured concentrations that are less than this limit are replaced with the minimum detection limit value and comment coded with a “B” in the variable code comment column. For example, the measured concentration of NO23F was 0.0005 mg/L as N (MDL=0.0008), the reported value would be 0.0008 with a “B” placed in the NO23F comment code column. Calculated parameters are comment coded with a “C” and if any of the components used in the calculation are below the MDL, the calculated value is removed and also comment coded with a “B”. If a calculated value is negative, the value is removed and comment coded with an “N”.

Note: The way below MDL values are handled in the NERRS SWMP dataset was changed in November of 2011. Previously, below MDL data from 2002-2006 were also coded with a B, but replaced with -9999 place holders. Any 2002-2006 nutrient/pigment data downloaded from the CDMO prior to December November of 2011 will contain -9999s representing below MDL concentrations.

Comment Code	Definition
A	Value above upper limit of method detection
B	Value below method detection limit
C	Calculated value
D	Data deleted or calculated value could not be determined due to deleted data, see metadata for details
H	Sample held beyond specified holding time
K	Check metadata for further details
M	Data missing, sample never collected or calculated value could not be determined due to missing data
P	Significant precipitation (reserve defined, see metadata for further details)
U	Lab analysis from unpreserved sample
S	Data suspect, see metadata for further details

Missing data

Two diel samples are missing for March due to the ISCO hose being clogged with algae which prevented water from being collected for the last two sample periods.

Analysis for phaeophytin was discontinued in July of 2006, when chlorophyll a analysis began in-house.

Explanation of comment codes

Unusually high amounts of PO₄ (0.0978 mg/L) were present for one grab sample taken at Loosin Creek on 15 February at 14:39 for unknown reasons. This value is considered suspect.

In March, there was a malfunction with the ISCO due to the hose getting clogged with algae. Bottle #9, filled at 5:43 on 16 March was completely full and bottle #10, filled at 7:54 was more full than usual.

March and June samples were held a few days beyond the standard five day period prior to mailing to VIMS for analysis. These samples were filtered and kept frozen according to protocol prior to shipping.

The diel sample collected at Research Creek on 17 May at 5:57 contained unusually high amounts of chl a (66.97 µg/L) for unknown reasons, but possibly due to macroalgae contamination. This value is considered suspect.

The following parameters had blanks that were above the minimum detection limit (MDL) during nutrient analyses (all values are reported in mg/L):

April NH₄: blank=0.0080, MDL=0.0054

June PO₄: blank=0.0019, MDL=0.0015

August NO₃: blank=0.0019, MDL=0.0010

August PO₄: blank=0.0017, MDL=0.0015

October NH₄: blank=0.0180, MDL=0.0054

November NH₄: blank=0.0273, MDL=0.0054

December PO₄: blank=0.0043, MDL=0.0015

The diel sample collected at Research Creek on 11 October at 6:12 contained unusually high amounts of PO₄ (0.1060 mg/L) for unknown reasons. This value is considered suspect.

The chlorophyll a samples for November may have samples switched from Research Creek and Zeke's Basin (samples ZB-D and RC-D) during processing. Previous data show that the Research Creek site has traditionally had lower chlorophyll a values than the Zeke's Basin site. Sample values for each site are suspect.

Chlorophyll data for a few of the ISCO samples seemed elevated during December. The analyst noticed dark filters and potential contamination by suspended macroalgae or detritus material. The samples were taken on 5 December at 23:24 and 6 December at 8:08 and 14:41; ISCO bottles #5,9 and 12 respectively.

15. QA/QC programs

a) Precision

- i) Field variability – NOCNERR collects two successive grab samples for the monthly grab sample program.
- ii) Laboratory variability – The VIMS Analytical Service Center for Nutrients analyzes a laboratory duplicate once for every ten samples.
- iii) Inter-organizational splits – none

b) Accuracy

- i) Sample spikes – The VIMS Analytical Service Center for Nutrients analyzed a matrix spike once for every ten samples.
- ii) Standard reference material analysis – none
- iii) Cross calibration exercises – none

16. Other remarks

On 5/19/2025 this dataset was updated to include embedded QAQC flags and codes for anomalous/suspect, rejected, missing, and below detection limit data. System-wide monitoring data beginning in 2007 were processed to allow for QAQC flags and codes to be embedded in the data files rather than using the original single letter codes used for the nutrient and pigment dataset along with the detailed sections in the metadata document for suspect, missing, and rejected data. Please note that prior to 2007, rejected data were deleted from the dataset so they are unavailable to be used at all. Suspect, missing, rejected and below minimum detection flags and appropriate three letter codes were embedded retroactively for dataset consistency. The QAQC flag/codes corresponding to the original letter codes are detailed below.

Flag/code	If also C	Historic Letter Code	Historic Code Definition
<1> [SUL]		A	Value above upper limit of method detection
<4> [SBL]	<4> [SCB]	B	Value below method detection limit
no need to flag/code unless combined		C	Calculated value
<3> [GCD]	<3> [GCR]	D	Data deleted or calculated value could not be determined due to deleted data, see metadata for details
<1> (CHB)		H	Sample held beyond specified holding time
<0> (CSM) unless other flag		K	Check metadata for further details
<2> [GDM]	<2> [GOM]	M	Data missing, sample never collected or calculated value could not be determined due to missing data
<3> [SNV] and <1> [SOC] for components (CRS) or F_Record (CRS)		N	Negative calculated value
<0> (CUS)		P	Significant precipitation (reserve defined, see metadata for further details)
<1> (CSM)		U	Lab analysis from unpreserved sample
		S	Data suspect, see metadata for further details

Tropical Depression Alberto passed over the area on 14 June 2006.

Tropical Storm Ernesto passed over the area on 31 August 2006.

An estimated 60,000 gallons of sewage spilled into Hewlett's creek (across the waterway from Loosin Creek) due to a leaking sewer pipe on February 25, 2006. The pipe may

have been leaking for months and the total amount of sewage spilled is unknown. Spills also occurred in July and November.

Samples from the following months were collected in less than 72 hours of a rain event: February, March, April, May, July (RC and LC grabs only), August, September, October, November and December