North Carolina National Estuarine Research Reserve (NOC)

NERR Nutrient Metadata Report

January – December 2005 Latest Update: May 19, 2025

I. Data Set and Research Descriptors

1. Principal investigator(s) and contact persons

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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 (NH4+), nitrate (NO3-), nitrite (NO2-), 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 shipment to the Virginia Institute for Marine Science (VIMS). For chlorophyll *a* analysis, volumes of 100 ml are filtered through a 47 mm Whatman GF/F glass microfiber filter (pore size 0.7 µm) 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. Samples are then filtered through a 47mm Millipore MF membrane filter (mixed cellulose ester, pore size 0.45µm). 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):

Site	Start Date	Start Time	End Date	End Time
RC	01/06/05	11:05	01/07/05	11:06
RC	02/08/05	13:25	02/09/05	11:15
RC	03/10/05	12:00	03/11/05	12:01
RC	04/11/05	16:12	04/12/05	16:13
RC	05/09/05	13:13	05/10/05	13:14
RC	06/07/05	14:15	06/08/05	14:16
RC	07/19/05	14:30	07/20/05	14:31
RC	08/16/05	09:55	08/17/05	09:56
RC	09/19/05	12:45	09/20/05	12:46
RC	10/17/05	13:20	10/18/05	13:21
RC	11/14/05	12:15	11/15/05	12:16
RC	12/19/05	14:00	12/20/05	14:01

Grab Sampling (All times in EST):

Site	Start Date	Rep 1 Time	Start Date	Rep 2 Time
LC	01/07/05	11:14	01/07/05	11:14
EC	01/07/05	09:05	01/07/05	09:06
ZB	01/07/05	09:51	01/07/05	09:52
RC	01/07/05	11:31	01/07/05	11:32
LC	02/08/05	12:25	02/08/05	12:25
EC	02/09/05	10:25	02/09/05	10:26
ZB	02/09/05	10:57	02/09/05	10:57
RC	02/08/05	12:54	02/08/05	12:54
LC	03/11/05	12:45	03/11/05	12:46
EC	03/11/05	10:45	03/11/05	10:45
ZB	03/11/05	10:14	03/11/05	10:15
RC	03/11/05	13:08	03/11/05	13:08
LC	04/11/05	15:29	04/11/05	15:30
EC	04/11/05	13:48	04/11/05	13:49
ZB	04/11/05	13:23	04/11/05	13:24
RC	04/11/05	15:53	04/11/05	15:54
LC	05/09/05	12:36	05/09/05	12:37
EC	05/09/05	10:26	05/09/05	10:27
ZB	05/09/05	11:48	05/09/05	11:49
RC	05/09/05	12:52	05/09/05	12:53
LC	06/07/05	13:35	06/07/05	13:36
EC	06/07/05	10:18	06/07/05	10:19
ZB	06/07/05	10:42	06/07/05	10:43
RC	06/07/05	14:00	06/07/05	14:01
LC	07/19/05	11:10	07/19/05	11:11
EC	07/19/05	09:00	07/19/05	09:01
ZB	07/19/05	08:26	07/19/05	08:27
RC	07/19/05	11:46	07/19/05	11:47
LC	08/17/05	09:53	08/17/05	09:54
EC	08/17/05	06:47	08/17/05	06:48
ZB	08/17/05	07:13	08/17/05	07:14
RC	08/17/05	10:12	08/17/05	10:13
LC	09/19/05	13:10	09/19/05	13:11
EC	09/19/05	10:03	09/19/05	10:04
ZB	09/19/05	10:37	09/19/05	10:38
RC	09/19/05	12:29	09/19/05	12:30
LC	10/17/05	12:25	10/17/05	12:26
EC	10/17/05	09:21	10/17/05	09:22
ZB	10/17/05	09:46	10/17/05	09:47
RC	10/17/05	12:49	10/17/05	12:50
LC	11/14/05	11:35	11/14/05	11:36
EC	11/14/05	08:58	11/14/05	08:59
ZB	11/14/05	09:24	11/14/05	09:25

RC	11/14/05	12:01	11/14/05	12:02
LC	12/20/05	15:17	12/20/05	15:18
EC	12/20/05	12:28	12/20/05	12:29
ZB	12/20/05	12:53	12/20/05	12:54
RC	12/20/05	14:56	12/20/05	14:57

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 it liability due to any losses resulting in any way from the use of this data.

NERR water quality 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. 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 2005.

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 Par	rameters:		
	Chlorophyll a	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	2005
Nitrite	NO2F	0.0002 mg/L as N	2005
Nitrate +	NO23F	0.0010 mg/L as N	2005
Nitrite			
Orthophosphate	PO4F	0.0015 mg/L as P	2005
Chlorophyll	CHLA_N	$0.5 \mu g/L$	2005
Phaeophytin	PHEA	$0.5 \mu\mathrm{g/L}$	2005

13. Laboratory methods

a) Parameter: Ammonia (NH4F)

Method References: Virginia Institute of Marine Science Analytical Service Center. U.S. EPA. 1974. <u>Methods for Chemical Analysis of Water and Wastes</u>, 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 \square m pore size, 47 mm diameter) using a vacuum pump. The pooled filtrate is then filtered through a Millipore membrane filter (0.45 \square m 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 + Nitirite (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-napthylenediamine dihydrochloride to form a reddish/purple azo dye and is read colorimetrical at 540 nm. Nitrate concentration is obtained by subtracting the corresponding nitrite value from the $NO_3^- + NO_2^-$ 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 \square m pore size, 47 mm diameter) using a vacuum pump. The pooled filtrate is then filtered through a Millipore membrane filter (0.45 \square m 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 \square m pore size, 47 mm diameter) using a vacuum pump. The pooled filtrate is then filtered through a Millipore membrane filter (0.45 \square m 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. <u>A Practical Handbook of Seawater Analysis</u>. 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 Fluoresence. Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Matrices Revision 1.2. September 1997.

<u>Using the Turner Designs Model 10 Analog, The 10AU Digital, Or the TD-700 Fluorometer with EPA Method 445.0</u>. 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 \square m 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	Definition
Code	
A	Value above upper limit of method detection
В	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
Н	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

One diel sample is missing in February due to misalignment of the sample bottles in the ISCO.

Two diel samples are missing in March due to the ISCO collection hose being out of the water at extreme low tides.

One diel sample is missing in September due to the ISCO collection hose being out of the water at extreme low tide.

Suspect data

Diel samples for July were not retrieved on 20 July due to severe thunder and lightning storms. The ISCO was retrieved on 21 July at 7:30. Samples were retained but considered suspect.

Quantities of NO23 (0.0039mg/L), PO4 (0.0033mg/L), and NO2 (0.0011mg/L) were found in the blank submitted to VIMS in September to test our filtering technique. September values for NO23, PO4, NO2, NO3, and DIN should be considered suspect.

Quantities of NH4 (0.0207mg/L) were found in the blank submitted to VIMS in November to test our filtering technique. November values for NH4 and DIN should be considered suspect.

There was a spike in NH4 values for the diel sample taken at 20:59 on 14 November for unknown reasons. Values for NH4 and DIN for this date and time should be considered suspect.

Quantities of NO23 (0.0028mg/L), PO4 (0.0061mg/L), and NH4 (0.0158mg/L) were found in the blank submitted to VIMS in December to test our filtering technique. December values for NO23, PO4, NO3, NH4 and DIN should be considered suspect.

December samples collected on 19 and 20 of December 2005 could not be shipped to VIMS until 3 January 2006 due to VIMS staff being away for the holidays.

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.

		Historic	
Flag/code	If also C	Letter Code	Historic Code Definition
<1>[SUL]		Α	Value above upper limit of method detection
<-4>[SBL]	<-4>[SOB]	В	Value below method detection limit
no need to flag/code unless combined		С	Calculated value
<-3>[GQD]	<>[CCR]	D	Data deleted or calculated value could not be determined due to deleted data, see metadata for details
<1>(OHB)		Н	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		N	Negative calculated value
(CRE) or F_Record (CRE)		Р	Sgnificant precipitation (reserve defined, see metadata for further details)
<0>(OUS)		U	Lab analysis from unpreserved sample
<1>(CSM)		S	Data suspect, see metadata for further details

Grab samples for February were taken on two different days, but still within a 24 hour period. Loosin Creek and Research Creek grab samples were collected on 8 February, while East Cribbings and Zeke's Basin samples were collected on 9 February.

Approximately 3 million gallons of sewage spilled into Hewlett's Creek (across the Intracoastal Waterway from Loosin Creek) on 1 July 2005.

September samples were collected five days after Hurricane Ophelia made landfall (14 September 2005), resulting in substantial rainfall to the area.

Samples from the following months were collected in less than 72 hours of a rain event:

March, June, August, December

Rainfall amounts are reported below:

2005 Daily, monthly, and annual precipitation totals

January Date

Daily Precip Totals (mm) 01/05/2005 0.3 01/13/2005 0.3 26.2 01/15/2005 01/17/2005 1.0 01/18/2005 0.3 01/23/2005 2.3 01/27/2005 0.5 01/30/2005 1.3 01/31/2005 9.7

Monthly Total (mm) 41.9

February

Date	Daily Precip Totals (mm)
02/04/2005	9.9
02/10/2005	2.5
02/14/2005	1.0
02/15/2005	0.3
02/21/2005	3.0
02/24/2005	4.8
02/27/2005	11.2
02/28/2005	6.6

Monthly Total (mm) 39.3

March

Date	Daily Precip Totals (mm)
03/05/2005	0.3
03/08/2005	4.3
03/14/2005	0.5
03/16/2005	11.9
03/17/2005	0.5
03/19/2005	1.5
03/20/2005	3.6
03/22/2005	11.9
03/23/2005	7.4
03/27/2005	9.1
03/28/2005	17.3
03/31/2005	0.3

Monthly Total (mm) 68.6

April

Date	Daily Precip Totals (mm)
04/01/2005	4.8
04/02/2005	23.6
04/07/2005	6.6
04/08/2005	8.6
04/13/2005	8.4
04/14/2005	18.8
04/26/2005	4.1

Monthly Total (mm) 74.9

May

Date	Daily Precip Totals (mm)
05/01/2005	3.0
05/05/2005	29.5
05/06/2005	38.9

```
      05/16/2005
      3.8

      05/17/2005
      11.2

      05/20/2005
      12.7

      05/21/2005
      7.4

      05/23/2005
      10.2

      05/24/2005
      13.5

      05/30/2005
      4.1
```

Monthly Total (mm) 134.3

June Daily Precip Totals (mm) Date 06/01/2005 5.8 06/02/2005 28.2 06/04/2005 15.5 06/07/2005 0.3 06/09/2005 0.5 06/11/2005 0.3 06/17/2005 1.3 06/25/2005 16.0 06/26/2005 0.5 06/28/2005 0.5

Monthly Total (mm) 90.7

21.8

06/29/2005

```
July
              Daily Precip Totals (mm)
Date
07/03/2005
              0.3
07/04/2005
              0.5
07/05/2005
              6.6
07/08/2005
              0.3
07/10/2005
              0.8
07/13/2005
              31.2
07/14/2005
              12.4
              9.9
07/15/2005
07/20/2005
              22.6
07/21/2005
              5.8
07/23/2005
              30.7
07/29/2005
              8.1
07/30/2005
              7.6
07/31/2005
              17.0
```

Monthly Total (mm) 153.8

August

Date	Daily Precip Totals (mm)
08/08/2005	1.5
08/09/2005	35.1
08/10/2005	7.4
08/15/2005	1.0
08/17/2005	3.8
08/18/2005	9.4
08/19/2005	45.0
08/20/2005	2.8
08/23/2005	73.2
08/24/2005	4.1
08/26/2005	0.3
08/27/2005	24.9

Monthly Total (mm) 208.5

September Date

Date	Daily Precip Totals (mm)
09/12/2005	5.8
09/13/2005	25.4
09/14/2005	111.8
09/15/2005	1.3
09/24/2005	0.3
09/25/2005	3.0

Monthly Total (mm) 147.6

October

October	
Date	Daily Precip Totals (mm)
10/05/2005	26.9
10/06/2005	35.6
10/07/2005	100.8
10/08/2005	65.5
10/10/2005	2.3
10/11/2005	0.5
10/12/2005	0.3
10/21/2005	0.3
10/24/2005	24.4
10/28/2005	4.6
10/30/2005	0.3
10/31/2005	0.3

Monthly Total (mm) 261.8

November

Date Daily Precip Totals (mm)

```
0.3
11/08/2005
11/09/2005
             0.3
11/15/2005
             1.3
11/16/2005
             0.5
11/17/2005
             1.3
11/20/2005
             0.8
11/21/2005
             75.7
11/22/2005
             1.0
11/27/2005
             6.9
11/28/2005
             1.5
11/29/2005
             6.4
11/30/2005
             0.3
```

Monthly Total (mm) 96.3

December

Decemen	
Date	Daily Precip Totals (mm)
12/04/2005	0.5
12/05/2005	1.8
12/06/2005	0.3
12/08/2005	0.8
12/09/2005	10.4
12/15/2005	14.0
12/17/2005	8.4
12/18/2005	28.7
12/28/2005	5.8
12/29/2005	5.6

Monthly Total (mm) 76.3

Annual Total (mm) 1394.0