North Carolina National Estuarine Research Reserve (NOC) Nutrient Metadata Report April – December 2002

Latest Update: May 19, 2025

- I. Data Set and Research Descriptors
- 1. Principal investigator(s) and contact persons
  - a) Reserve contacts

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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 NOCNERR. Water samples were collected, processed, and analyzed for the following parameters: ammonium (NH4+), nitrate (NO3-), nitrite (NO2-), ortho-phosphate, and Chla.

#### a) Monthly Grab

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

### b) Diel Sampling Program

Once per month, samples were 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

### a) Monthly Grab Sampling Program

Monthly grab samples were taken at three stations within the Masonboro Island and Zeke's Island components of the NOCNERR. Samples were taken at three of the principal NOCNERR datasonde stations, which are Loosin Creek, Zeke's Basin and East Cribbings. Efforts were 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 were made between neap and spring tide conditions. Replicate (N=2) samples were collected by hand at an approximate depth of 10 cm. All samples were collected in amber, widemouth, nalgene sample bottles that were previously acid washed (10%), rinsed (3x) with distilled-deionized water, dried and followed by rinsing (3x) of ambient water prior to collection of the sample. Samples were immediately placed on ice, in the dark and returned to the laboratory. Once in the laboratory, samples were shaken and processed for nutrient and Chla analysis.

## b) Diel sampling Program

Diel samples were taken at the Research Creek datasonde station. Samples were collected over a 24 hour time period at intervals of two hours and eleven minutes using an ISCO auto-sampler. Samples were collected at a fixed depth (0.5m) from the bottom which reflected the water mass sampled by the datasonde. No distinction was made between neap and spring tide conditions. Efforts were made to allow for an antecedent dry period of 72 hours prior to sampling. All samples were collected in 1000 ml polyethylene bottles that were previously acid washed (10% HCL), rinsed (3x) with distilled-deionized water and dried. Samples were stored inside the ISCO sampler and kept cool with ice. Once in the laboratory, samples were shaken and processed for nutrient and Chla analysis.

#### 4. Site location and character

The components of North Carolina's National Estuarine Research Reserve (from north to south) are: the Northern Sites component, Rachel Carson, Masonboro Island, Zeke's Island, and the Southern Sites component. They are located along the southeastern coast of the United States in the Atlantic Ocean. Currently, only data from Masonboro Island and Zeke's Island components are transferred to the CDMO.

#### a) Research Creek, Masonboro Island

The first Masonboro Island site 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 has a salinity range from 18-35 ppt and a tidal range that averages 1.7 meters. The creek bottom is characterized by sand and detritus based sediment with areas of soft mud with a depth ranging from 0.34 to 2.45 m. The sole source of freshwater is rain. 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 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 has a salinity range from 18-35 ppt and a tidal range that averages 1.7 meters. 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. The sole source of freshwater is rain. 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 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 has a salinity range from 15-35 ppt and a tidal range that averages 2 meters. Depth varies, but typically it can be found to range from 0.11 to 2.30 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; not accessible to road traffic and minimal boat traffic.

#### d) Zeke's Basin, Zeke's Island

The second Zeke's Island site 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 salinity range from 11-35 ppt and a tidal range that averages 2 meters. Depth varies, but typically it can be found to range from 0.1 to 2 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; not accessible to road traffic and minimal boat traffic.

# 5. Coded Variable Code Definitions

Nutrient Sampling Stations: (noclcnut) Loosin Creek, (nocecnut) East Cribbing, (nocrcnut) Research Creek, and (noczbnut) Zeke's Basin

Monitoring Programs:

- (1) Grab Sampling
- (2) Diel Sampling

# 6. Data collection period

# Diel Sampling:

Site	Start Date	Start Time	End Date	End Time
RC	04/22/02	10:00	04/23/00	10:00
RC RC	05/22/02 06/26/02	10:00 03:00	05/23/00 06/27/02	10:01 03:01
RC	07/22/02	12:19	07/23/02	12:20
RC	08/21/02	12:49	08/22/02	12:50
RC	09/24/02	09:15	09/25/02	09:16
RC	10/21/02	09:45	10/22/02	09:46
RC	11/18/02	12:00	11/19/02	12:01
RC	12/16/02	11:45	12/17/02	11:46

# Grab Sampling:

Site	Start Date	Start Time	End Date	End Time
LC	04/23/02	10:40	04/23/02	10:40
EC	04/23/02	15:05	04/23/02	15:05
ZB	04/23/02	15:35	04/23/02	15:35
LC	05/23/02	10:00	05/23/02	10:00
EC	05/24/02	08:50	05/24/02	08:50
ZB	05/24/02	09:20	05/24/02	09:20
LC	06/27/02	09:00	06/27/02	09:00
EC	06/24/02	10:14	06/24/02	10:14
ZB	06/24/02	10:35	06/24/02	10:35
LC	07/22/02	10:10	07/22/02	10:10
EC	07/24/02	09:15	07/24/02	09:15
ZB	07/24/02	09:40	07/24/02	09:40
LC	08/22/02	15:03	08/22/02	15:03

EC	08/26/02	10:34	08/26/02	10:34
ZB	08/26/02	11:31	08/26/02	11:31
LC	09/24/02	09:00	09/24/02	09:00
EC	09/24/02	10:25	09/24/02	10:25
ZB	09/24/02	10:40	09/24/02	10:40
LC	10/21/02	09:47	10/21/02	09:47
EC	10/23/02	11:50	10/23/02	11:50
ZB	10/23/02	12:06	10/23/02	12:06
LC	11/18/02	10:30	11/18/02	10:30
EC	11/21/02	11:19	11/21/02	11:19
ZB	11/21/02	11:41	11/21/02	11:41
LC	12/16/02	09:45	12/16/02	09:45
EC	12/17/02	09:25	12/17/02	09:25
ZB	12/17/02	09:40	12/17/02	09:40

Note: Time is coded based on a 2400 hour clock and is referenced to Eastern Standard Time (EST).

# 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 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 Chla data files, in excel format, were sent to NOCNERR by the VIMS Analytical Service Center for Nutrients. Files consist of sampling station ID, date and parameter values expressed in unit concentrations. The research associate verified all parameter values in the excel file through cross comparison with laboratory data files. Missing data were verified through inspection of field logs, inserted into the data files and denoted by a blank cell by Paula May, NOCNERR research associate.

### 10. Parameter Titles and Variable Names by Data Category

Data Category	Parameter	Variable Name Units	s of Measure			
i) Phosphorus:	Orthophosphate	PO4F	mg/l as P			
ii) 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			
iii) Other Lab Parameters:						
	Chlorophyll a	CHLA_N	μg/l			
	Phaeophytin	PHEA	μg/l			

#### Notes:

- 1. Time is coded based on a 2400 hour clock and is referenced to Eastern Standard Time (EST).
- 2. Reserves have the option of measuring either NO23 or NO2 or NO3.

### 11. Measured and Calculated Laboratory Parameters

# a) Variables Measured Directly

Nitrogen species: NO2F, NO23F, NH4F

Phosphorus species: PO4F Other: CHLA\_N

### b) Computed Variables

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 MDL's; 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	0.0015 mg/l as	N	2002
Nitrite	NO2F	0.0008 mg/l as	N	2002
Nitrate + Nitrite	NO23F	0.0008 mg/l as	N	2002
Orthophosphate	PO4F	0.0006 mg/l as	Р	2002
Chlorophyll	CHLA	0.5 μg/l	2002	
Pheophytin	PHEA	0.5 μg/l	2002	

# 13. Laboratory Methods

#### a) Parameter: NH4

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 37oC and is measured colorimetrically at 660 nm. The range is 0.01 - 2.0 mg/L.

Preservation Method: Sample is filtered through a 0.45 um disposable disk filter and stored at -20°C until analyzed.

### b) Parameter: NO23, NO3, and NO2

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 NO3- + NO2- concentration. The color development chemistry is the same as that used in Nitrite. Range is 0 -1.2 mg/L.

Preservation Method: Sample is filtered through a 0.45 um disposable disk filter and stored at -20°C until analyzed.

#### c) Parameter: 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 catalizyed at 40?C and measured colorimetrically at 880nm. The range is 1-50 ppb.

Preservation Method: Sample is filtered through a 0.45 um disposable disk filter and stored at -20°C until analyzed.

### d) Parameter: CHLA, 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 Fluoresence. 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 4oC, 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 -20oC for a few days is acceptable. If pheophytin measurements are desired, the sample is acidified and read again.

Preservation Method: A known volume of sample is filtered onto a 25 mm GF/F filter, folded in half and wrapped in aluminum foil. Foil is stored at -20°C until analyzed.

14. Reporting of Missing Data, Data with Concentrations Lower than Method Detection Limits and Other Flagged Data

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	
Α	Value above upper limit of method detection
В	Value below method detection limit
С	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
Р	Significant precipitation (reserve defined, see metadata for
	further details)
U	Lab analysis from unpreserved sample
S	Data suspect, see metadata for further details

### 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.
- 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>[SCB]	В	Value below method detection limit
no need to flag/code unless combined		С	Calculated value
<-3>[GQD]	<>[GCR]	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>(C3M) unless other flag		K	Check metadata for further details
<-2>[GDM]	<-2>[GOM]	М	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	Significant precipitation (reserve defined, see metadata for further details)
<0\(CUS)		U	Lab analysis from unpreserved sample
<1>(CSM)		S	Data suspect, see metadata for further details

Explanation for the occurrences of code "K"

11/21/02 ZB Results for PO4F, NO23F, NO2F, and NH4F were not reported from VIMS April diel samples were collected every two hours by the ISCO rather than 2 hours and 11 minutes. The ISCO did not collect at 8:00 because of a power failure, so the last sample was collected at 10:00 instead.

The following grab samples were collected during a flood tide rather than an ebb tide:

04/23/02 EC and ZB 06/27/02 LC 08/22/02 LC and EC 09/24/02 LC

Samples from the following months were collected in less than 72 hours of a rain event: June, July, August, September, October, and November.

Rainfall amounts for these months are reported below:

June		(mm)
7	4.572	
14	19.558	
17	1.016	

```
18
        59.944
19
        1.270
20
        .254
21
        5.334
22
        40.640
23
        1.524
26
        1.016
27
        22.606
        1.778
28
        5.080
29
July
                   (mm)
5
        6.096
6
        3.302
10
        11.938
        14.478
11
13
        7.366
14
        30.988
15
        13.970
20
        1.270
21
        .762
23
        24.130
24
        1.524
        8.128
25
26
        7.874
       8.890
31
                    (mm)
August
4
        .762
6
        5.334
        .254
16
19
        2.032
24
        11.684
29
        8.382
30
        48.260
31
        72.898
September
                     (mm)
1
        24.130
2
        10.922
```

6

7

4.318

.762

9	10.668
10	5.842
12	.254
14	8.890
15	13.716
16	5.334
23	13.462
24	13.716
26	6.604
27	4.064
28	13.208

October		(mm)
10	5.080	
11	23.622	
13	4.064	
14	.762	
15	23.368	
21	16.002	
22	8.128	
24	16.002	
28	3.302	
29	.254	
30	1.270	

	(mm)
1.524	
2.032	
10.922	
1.524	
3.810	
4.318	
10.160	
7.112	
22.098	
7.620	
	2.032 10.922 1.524 3.810 4.318 10.160 7.112 22.098