Reserve Name (include 3 letter code here) SAP NERR Nutrient Metadata

Months and year the documentation covers: January 1, 2022 - December 31, 2022

Latest Update: Date that the last edits were made 6/14/23

Note: This is a provisional metadata document; it has not been authenticated as of its download date. Contents of this document are subject to change throughout the QAQC process and it should not be considered a final record of data documentation until that process is complete. Contact the CDMO (cdmosupport@baruch.sc.edu) or reserve with any additional questions.

I. Data Set and Research Descriptors

1) Principal investigator(s) and contact persons -

[Instructions/Remove: List the reserve staff members responsible for the implementation and collection of the nutrient data. List the laboratory staff members responsible for processing of the samples and data output. Include name, title, mailing address, phone number, and email address for the Research Coordinator, SWMP technician(s), person(s) responsible for data management, and laboratory contact.]

A) Reserve Contact

a. Rachel Guy (Research Coodinator) rachel.guy@dnr.ga.gov 1766 Landing Rd SE Darien, GA 31305 912-262-3173

B) Laboratory Contact

a. Carol Pollard (VIMS Lab Manager)
pollard@vims.edu
Rt 1208 Greate Rd Gloucester Point, VA 23062
804-684-7213

C) Field Contacts (SWMP Managers)

 a. Thompson Rose (1/2022 – 3/2022) douglas.rose@dnr.ga.gov 912-577-7724
 b. Ivanna Knox (8/2022 – 12/2022)

ivanna.knox@dnr.ga.gov

2) Research objectives -

[Instructions/Remove: Describe briefly the nature of each monitoring program resulting in this data set (monitoring along land use, vertical, salinity or habitat gradients).]

The nutrient monitoring program is designed upon spatial deployment across a wide variety of marsh types with differing fresh and marine water mixing. These differing dynamics allow scientists and researchers to select from both a wide variety of research sites as well as tailor research programs to specific tidal dynamics and utilize the Reserves SWMP data acquisitions to the maximum extent. Additionally, from a long-term trend perspective the variety of marsh types and hydrology being monitored will allow for a better understanding of the different effects of sea-level rise upon marsh type. Due to a lack of residential development and very low human activity within the watersheds of the sites, they serve as a proxy for reference conditions with the various marsh and associated hydrology types for the creeks and river stations. All of the sites selected have very little anthropogenic nutrient influences. The following brief descriptions are associated with each nutrient monitoring site. For more detail please refer to the site descriptors located under section (4) of this document and/or contact the Research Coordinator at the SAP NERR for detailed information of any/all sites.

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Lower Duplin: Located at the mouth of the Duplin River with large, rapid and near-complete hydraulic exchange with Doboy Sound within each diurnal cycle. Typical of a high salinity, well mixed estuary site.

Hunt Dock: Located on the upper Duplin with relatively high hydraulic retention requiring an estimated 6-7 diurnal events to complete a total hydraulic exchange. Rainfall may drop salinity precipitously in the basin depending on tidal height, duration and volume of precipitation.

Cabretta Creek: Located on the eastern side of Sapelo Island with direct exchange with the Atlantic Ocean. Creek is typical of high salinity, high oceanic exchange and near complete hydraulic exchange with each diurnal event. Creek is extremely buffered from rainfall (event driven) fluctuations in salinity.

Dean Creek: Located on the southern end of Sapelo is the primary drainage of the inter-dune (located amid primary and secondary dune systems) meadow. This site is highly susceptible to very high salinity fluctuations associated with rainfall events on both seasonal and short—term, event driven scales. Tidal exchange is complete at each diurnal event and exchange water genesis is the Doboy Sound.

The Duplin River is a tidal basin with no freshwater influence within its headwaters apart from surficial aquifer weeping from the perched lens of water associated with Sapelo Island. This nutrient monitoring effort is tied into the Georgia Coastal Ecosystems, Long-Term Ecological Research (GCE-LTER) initiative and the University of Georgia Marine Extension Service water quality database whose collection and analysis of the water samples facilitates the database. This long-term data set is being developed to provide information on estuarine water mixing within the well-studied Duplin River basin in addition to providing a long-term characterization of water quality as related to nutrient loading within the Duplin River

- a) Monthly grab sampling program The Monthly Grab Sampling Program focuses on documentation of baseline reference nutrient trends within a wide array of local marsh systems with differing hydrology.
- b) Diel sampling program (mention if samples were taken over a lunar day) The Diel Sampling Program focuses on short-term temporal variability over a lunar tidal cycle.

3) Research methods -

[Instructions/Remove: Detail the specifies of sample collection, collection intervals, sample processing, how samples are held, and QAQC of the equipment and analyzers for each program.]

a) Monthly grab sampling program

Monthly grab samples were taken at four stations within the Duplin River estuary from January to December 2022. Bottom water samples were taken at the Lower Duplin (LD), Hunt Dock (HD), Cabretta Creek (CA) and Dean Creek (DC) stations using a Niskin style sampling bottle. All grab samples were taken sequentially in duplicate beginning near the time the last diel sample was collected by the ISCO sampler (this time corresponds to low tide at the end of the tidal cycle). Chronological collection times for each of the four sites vary. At the time of sample collection, latitude, longitude, time and depth were recorded. The depths at Cabretta and Dean Creek sites were estimated as sampling took place from a bridge. Samples collected were immediately placed on ice, in the dark and delivered to the Marine Extension Service laboratory for processing within six hours. Once in the laboratory,

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samples were filtered, frozen at -4°C and processed within the specified times (unless flagged) for nutrient and chlorophyll-a concentrations.

Processing each sample:

a) Using filter towers (acid-washed towers with a 0.45 um polycarbonate filter for nutrient filtering and clean towers with a GF/F filter for chlorophyll filtering), a small amount of sample was used to rinse the nutrient filter tower equipped with a filter and then the filtrate was discarded. The tower was then filled to the 250-mL mark. The chlorophyll tower with the GF/F filter was also filled to the 250-mL mark (or 500-mL mark if a larger filtration apparatus was used) and the towers were connected by small piece(s) of tubing. The vacuum pump was turned on to pull the sample through each filter and then the vacuum was released. The nutrient sample tower was disconnected and an acid-washed 250-mL polypropylene bottle was rinsed and filled with the filtrate. Space was left in the sample bottle for expansion during freezing at approximately -18 degC. If the first 250 or 500 milliliters went through the chlorophyll filter easily, the filtrate was discarded and an additional 50, 100, 250 or 500 milliliters was filtered, depending on suspended sediment load, to concentrate the sample onto the filter. The chlorophyll filter was then removed with tweezers and placed face up in a petri dish, wrapped in aluminum foil and labeled with the volume filtered and sample information. The chlorophyll filter towers were rinsed between replicate grabs with distilled water and the nutrient filter tower was acid-washed and DI water rinsed between samples.

b) Diel sampling program

As of November 2013, Reserve staff have been conducting all field work associated with this project. The recommended procedures for diel scheduling and sampling are as follows: www.usharbors.com Old Tower site, Sapelo Island was used to estimate low tide. As close to an early, low, neap tide as possible was selected each month for sampling. The ISCO sampler was deployed at the Lower Duplin (LD) site on the day previous to the grab sampling date chosen for that particular month with the sample line suction tube placed 1.5 feet below the surface of the water. The ISCO sampler collected the first diel sample as close as possible to the low tide predicted for the following day and continued collecting samples every two hours for the next 24 hours, representing a full tidal cycle and a total of 13 samples, ending at low tide near to the time when grab sampling began. The ISCO was turned off at the end of the collection period and the samples were secured with caps upon arriving at the site. The samples were filter processed in the laboratory by UGA Marine Extension laboratory personnel. The filtration process for the diel samples follows the same process as for grab samples described above. High-density polypropylene bottles were used to store the samples after filtration. Polypropylene bottles and filter towers were soaked in 10% HCl in preparation for the fieldwork, and then triple rinsed with distilled water. A squeeze bottle was used to acid wash (then rinse with distilled water) beakers and filter towers between filtering of each sample.

4) Site location and character –

[Instructions/Remove: Describe your NERR site in general and the sampling sites associated with each YSI data logger / nutrient collection in more detail. <u>Include the following</u> in your description for each sampling location. If certain characteristics apply to all sample sites or the entire reserve they may be discussed in an overview:]

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The Sapelo Island National Estuarine Research Reserve is located on the Southeastern Atlantic coast of the United States in McIntosh County, Georgia. The study area encompasses the Duplin River estuary, a tidally flushed drainage system flowing into Doboy Sound from the north and two inland creeks, Cabretta and Dean Creek. The Duplin River watershed occupies most of the Reserve, which also contains various forest types, sand dunes, a section of ocean beach and minor developed areas. The Duplin River estuary covers 3,300 acres between Sapelo Island and the mainland in McIntosh County. It drains a tidal bay and an extensive network of salt marshes about 6 miles long, into which there is little upland run-off. Diverse estuarine wetlands provide extensive and complex habitat types for fish and wildlife. The island contains several small, interior brackish and freshwater marshes fed by surficial aquifer expression (interdune meadow of Nannygoat beach: south end) and anthropogenic upland ditches and dikes produced in the early 19th century (north end). The upland forests are composed of several diverse habitats including long leaf pine/slash pine forests, climax maritime forests, small amounts of pond cypress bays and naturally regenerated loblolly pine forests which are timbered on a 70 year selectively cut harvest rotation. There are no current studies on pollutants in this area. Sapelo Island is typically considered a pristine environment, with minimal pollutant input.

Latitude and Longitude-

Lower Duplin: 31°25'04.3"N 81°17'45.4"W Hunt Dock: Lat: 31°28'43.3"N 81°16'23.2"W Cabretta Creek: 31°26'19.2"N 81°14'19.5"W Dean Creek: 31°23'41.2"N 81°16'11.5"W

Water Quality site descriptions:

Salinities at all Duplin River sites vary according to localized rainfall and associated runoff. The upper Duplin River site (Hunt Dock) experiences slightly lower salinities associated with rainfall events (2 -3ppt) as compared to the lower Duplin River site. Average salinities range from 15 ppt to 30 ppt depending on seasonal or event rainfall. Average tidal range of diurnal tidal cycle is approximately 2.5 meters twice daily. Due to high turbidity, all Duplin River sites are lacking any persistent submerged aquatic vegetation and have an unconsolidated sandy/mud bottom (soft sediment) typical of southeastern near-ocean estuaries. Marsh sediments are relatively pristine and free of pollutants based on sediment analysis conducted in 1996 by C. Alexander, Skidaway Institue of Oceanography. Watershed is dominated by oceanic tidal influences associated with Doboy Sound. Depths are as follows: Lower Duplin (LD) ranges from 1.5 meters to 6.0 meters depending on tide, and the Hunt Dock site maximum depth is 4.27 meters.

Cabretta Creek is fed directly from waters of the Atlantic Ocean. Cabretta experiences a maximum tidal range of approximately 4.3 meters. Average mean low water depth at the sample site is approximately 3.25 meters. Salinity ranges, with exception to major, long-term precipitation events, from 15-36 ppt, seasonally. The station is located on a small (one-lane), wooden, roadway bridge spanning Cabretta Creek, located on the island's extreme eastern side. The benthos is composed primarily of sand substrate with small, intertidal oyster reef conglomerate communities. Adjacent to the site is extensive, intertidal, bank stabilization (armoring) in the form of woven rip-rap fencing and granite rocks. This manipulation is slowly becoming stabilized via oyster reef community colonization. The adjacent marshes are dominated by Spartina alterniflora with occasional Juncus romerianus in the nearby fringe community habitat. The creek has very little adjacent uplands due to: 1) the low elevational gradient and 2) the area's geologically recent accretion genesis (Holocene)

resulting in sandy soils; of which neither condition allows for extensive floral colonization or stabilization.

The Dean Creek site is located on a steel bridge spanning Dean Creek, in close proximity to the adjacent Nannygoat Beach causeway. Dean Creek is a small tidal basin fed from the waters of Doboy Sound, which is located on Sapelo Island's south end. With exception to short duration local or long duration regional precipitation events, the creek's salinity normally ranges between 20 and 30 ppt. The benthic community consists of a sandy-mud substrate with occasional small, intertidal oyster reef community and mean tidal amplitude of approximately 8 feet. Average mean low water depth at the sample site is approximately 1 meter, but fluctuates due to bank erosion. The small creek feeds approximately 150 acres of Spartina alterniflora dominated salt marsh, which is interspersed with small 0.5-1 acre hammocks and salt pans. Fringe community components range from Loblolly pine forests with a sub-canopy of Yaupon holly to Wax myrtle and Sable Palm.

- a) latitude and longitude
- b) tidal range
- e) salinity range
- d) type and amount of freshwater input
- e) water depth (mean depth or depth range at site, NOT depth of sonde deployment)
- f) bottom habitat or type (soft sediment, grassbed, oyster bar, etc)
- g) pollutants in area
- h) description of watershed draining site

All_freserve name] SAP NERR historical nutrient/pigment monitoring stations:

Station Code	SWMP Status	Station Name	Location	Active Dates	Reason Decommissioned	Notes
CA	P	<u>Cabretta</u>	31°26'19.2"N 81°14'19.5"W	mm/dd/yyyy current04/2004 - current	NA	NA
DC	<u>P</u>	Dean Creek	31°23'41.2"N 81°16'11.5"W	<u>05/2004 - current</u>	<u>NA</u>	NA *
HD	<u>P</u>	Hunt Dock	31°28'43.3"N 81°16'23.2"W	07/1999 - current	<u>NA</u>	NA *
<u>LD</u>	<u>P</u>	<u>Lower</u> <u>Duplin</u>	31°25'04.3"N 81°17'45.4"W	01/1999 - current	<u>NA</u>	NA •
ML	<u>S</u>	Marsh Landing	31deg 25' 04.23" N, 81deg 17' 46.30" W	05/1995 – 12/1998	Site character	near surface deployment and the fouling with such a setup was too severe to harvest reliable data
FL	<u>S</u>	Flume Dock	31deg 28' 53.85"N 81deg 16"12.37"W	01/1995 – 12/1998	<u>NA</u>	<u>NA</u>

5) Coded variable definitions –

[Instructions/Remove: Explain the station code names and monitoring program codes. Use the following format:]

LD = Lower Duplin

 $\underline{HD} = \underline{Hunt Dock}$

CA = Cabretta Creek

DC = Dean Creek

Each individual sample is given a 3 part name code in addition to other codes. The 3 part name code, "sapldnut" for example, gives the reserve name (sap = Sapelo), station name (LD = Lower Duplin, etc), and SWMP program code (nut = nutrient monitoring program).

Sampling Site codes:

sapldnut - Sapelo Island nutrient data for Lower Duplin

saphdnut - Sapelo Island nutrient data for Hunt Dock

<u>sapcanut – Sapelo Island nutrient data for Cabretta Creek</u>

sapdcnut - Sapelo Island nutrient data for Dean Creek

The monitoring codes are set as "1" to indicate grab samples and "2" to indicate diel samples.

Replicates are also given specific codes. Grab samples in which duplicate field samples are taken

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utilize a "1" for the first sample and a "2" for the second sample. Subsequent lab splits of each field rep are labeled with an "S". Diel samples are always labeled with a "1" for the first lab replicate and an "S" for the second lab replicate. Only one actual sample is taken at each interval with the ISCO sampler.

ebytenut = Chesapeake Bay Virginia Taskinas Creek nutrients
...
monthly grab sample program = 1
diel grab sample program = 2

6) Data collection period -

[Instructions/Remove: List the date and time each sample was collected organized by station. For grab samples include replicate times or a general statement about the time frame for replicate collection. For diel samples, include start and end times for the sampling session.]

Diel Samp		times for the san	ilpiilig session-	
_	l, Dates/Time	<u>es</u>		_
Location	Start Date	Start Time	End Date	End Time
<u>LD</u>	1/10/2022	<u>11:00</u>	1/11/2022	<u>11:00</u>
<u>LD</u>	2/9/2022	<u>11:00</u>	2/10/2022	<u>11:00</u>
<u>LD</u>	3/24/2022	<u>11:00</u>	3/25/2022	<u>11:00</u>
<u>LD</u>	4/25/2022	<u>11:00</u>	4/26/2022	<u>11:00</u>
<u>LD</u>	<u>MISSING</u>	<u>MISSING</u>	<u>MISSING</u>	<u>MISSING</u>
<u>LD</u>	6/23/2022	<u>MISSING</u>	6/24/2022	<u>MISSING</u>
LD	<u>MISSING</u>	<u>MISSING</u>	<u>MISSING</u>	<u>MISSING</u>
<u>LD</u>	<u>MISSING</u>	<u>MISSING</u>	<u>MISSING</u>	<u>MISSING</u>
<u>LD</u>	9/19/2022	<u>10:30</u>	9/20/2022	<u>10:30</u>
<u>LD</u>	10/19/2022	<u>10:00</u>	10/20/2022	<u>10:00</u>
LD	11/22/2022	<u>10:30</u>	11/23/2022	<u>10:30</u>
<u>LD</u>	12/21/2022	<u>11:00</u>	12/22/2022	<u>11:00</u>

Grab Sampli	ing:							
Dates; Time					-	-	-	_
<u>Date</u>	LD1	LD2	HD1	HD2	CA1	CA2	DC1	DC2
1/11/2022	11:32	11:36	10:53	<u>10:58</u>	10:31	<u>10:38</u>	<u>9:59</u>	<u>10:03</u>
2/10/2022	<u>11:41</u>	<u>11:45</u>	<u>11:01</u>	<u>11:06</u>	<u>10:27</u>	<u>10:32</u>	<u>9:51</u>	<u>9:58</u>
3/25/2022	11:01	<u>11:07</u>	10:33	10:37	10:09	<u>10:13</u>	<u>9:38</u>	<u>9:42</u>
4/25/2022	11:36	11:37	<u>11:12</u>	<u>11:13</u>	10:46	<u>10:51</u>	<u>10:15</u>	<u>10:16</u>
MISSING								
6/24/2022	<u>11:35</u>	<u>11:40</u>	10:43	<u>10:45</u>	<u>10:07</u>	<u>10:10</u>	<u>9:30</u>	<u>9:32</u>
7/12/2022	12:30	12:35	<u>11:30</u>	<u>11:35</u>	10:55	<u>11:00</u>	<u>10:15</u>	<u>10:20</u>
8/19/2022	12:17	12:22	10:42	<u>10:48</u>	10:08	<u>10:13</u>	<u>9:20</u>	<u>9:25</u>
9/20/2022	<u>12:02</u>	<u>12:07</u>	<u>11:34</u>	<u>11:39</u>	<u>11:04</u>	<u>11:09</u>	<u>10:07</u>	<u>10:12</u>
10/20/2022	<u>11:59</u>	<u>12:04</u>	<u>11:18</u>	<u>11:23</u>	<u>10:50</u>	<u>10:55</u>	<u>10:12</u>	<u>10:17</u>
11/23/2022	<u>11:20</u>	<u>11:25</u>	<u>10:45</u>	<u>10:50</u>	<u>10:16</u>	<u>10:21</u>	<u>9:42</u>	<u>9:47</u>
12/22/2022	<u>11:00</u>	<u>11:05</u>	<u>10:25</u>	<u>10:30</u>	<u>10:00</u>	<u>10:05</u>	<u>9:20</u>	<u>9:25</u>

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7) Associated researchers and projects-

[Describe briefly other research (data collection) that correlates or enhances the nutrient data. You may provide links to other products or programs. At a minimum, mention the SWMP MET and WQ datasets as below.]

As part of the SWMP long-term monitoring program, XXX-SAP NERR also monitors 15-minute meteorological and water quality data which may be correlated with this nutrient/pigment dataset. These data are available at www.nerrsdata.org.

8) Distribution -

[Instructions/Remove: This section will address data ownership and data liability by including the following excernt.]

NOAA retains the right to analyze, synthesize and publish summaries of the NERRS Systemwide Monitoring Program data. The NERRS retains the right to be fully credited for having collected and processed the data. Following academic courtesy standards, the NERR site where the data were collected should be contacted and fully acknowledged in any subsequent publications in which any part of the data are used. The data set enclosed within this package/transmission is only as good as the quality assurance and 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 or 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.

Requested citation format:

NOAA National Estuarine Research Reserve System (NERRS). System-wide Monitoring Program. Data accessed from the NOAA NERRS Centralized Data Management Office website: www.nerrsdata.org; accessed 12 October 2022.

Also include the following excerpt in the metadata to address how and where the data can be obtained.

NERR nutrient data and metadata can be obtained from the Research Coordinator at the individual NERR site (please see Principal investigators and contact persons), from the Data Manager at the Centralized Data Management Office (please see personnel directory under the general information link on the CDMO home page) and online at the CDMO home page www.nerrsdata.org. Data are available in comma separated version format.

II. Physical Structure Descriptors

9) Entry verification -

[Instructions/Remove: This section explains how data acquisition, data entry, and data verification (QAQC) were performed before data were sent to the CDMO to be archived into the permanent database. Describe how your reserve receives data from the analytical laboratory, how it is entered into Excel, and how it is verified. If your reserve converts nutrient values to attain the required units of measurement, note that here and detail your process. List who was responsible for these tasks and include the following statement.]

Nutrient data are entered into a Microsoft Excel worksheet and processed using the NutrientQAQC Excel macro. The NutrientQAQC macro sets up the data worksheet, metadata worksheets, and MDL worksheet; adds chosen parameters and facilitates data entry; allows the user to set the number of significant figures to be reported for each parameter and

rounds using banker's rounding rules; allows the user to input MDL values and then automatically flags/codes measured values below MDL and inserts the MDL; calculates parameters chosen by the user and automatically flags/codes for component values below MDL, negative calculated values, and missing data; allows the user to apply QAQC flags and codes to the data; produces summary statistics; graphs selected parameters for review; and exports the resulting data file to the CDMO for tertiary QAQC and assimilation into the CDMO's authoritative online database.

[Example of conversion documentation, update for your laboratory results]. The University of Washington Marine Chemistry Laboratory calculates and reports results in µM. For purposes of consistency in the NERR System, Padilla Bay NERR calculates the concentrations as mg/1 1 based on atomic weights of 14.01, 30.97, 28.09, and 12.01 for N, P, Si, and C respectively. Therefore, Padilla Bay NERR staff multiplies the concentrations reported by the University of Washington Marine Chemistry Laboratory by 0.01401, 0.03097, 0.02809, and 0.01201 to yield concentrations in mg/L as N, P, Si, and C respectively.

10) Parameter titles and variable names by category -

[Instructions/Remove: Only list those parameters that are reported in the data. See Table 2 in the "Nutrient and Chlorophyll Monitoring Program and Database Design" SOP version 1.8 (March 2017) for a full list of available parameters. If NO2 and NO3 are not reported, modify note 2 to explain why.]

Required NOAA NERRS System-wide Monitoring Program nutrient parameters are denoted by an asterisk "*"

Data Category	Parameter	Variable Name	Units of Measure
Phosphorus and	d Nitrogen:		
1	*Orthophosphate	PO4F	mg/L as P
	*Ammonium, Filtered	NH4F	mg/L as N
	*Nitrite, Filtered	NO2F	mg/L as N
	*Nitrate, Filtered	NO3F	mg/L as N
	*Nitrite + Nitrate, Filtered	NO23F	0.
	Dissolved Inorganic Nitrogen	DIN	mg/L as N
Plant Pigments:	0 0		0,
0	*Chlorophyll a	CHLA_	_N μg/L
	Phaeophytin	PHEA	μg/L
Carbon:	1 7		1 0
Other Lab Para	.meters:		
	Silicate, Filtered	SiO4F	mg/L as SI
Microbial:	,		Ο,
Field Parameter	131		
	Water Temperature	WTEM	N °C

Notes:

- 1. Time is coded based on a 2400 clock and is referenced to Standard Time.
- 2. Reserves have the option of measuring either NO2 and NO3 or they may substitute NO23 for individual analyses if they can show that NO2 is a minor component relative to NO3.

11) Measured or calculated laboratory parameters –

[Instructions/Remove: This section lists all measured and calculated variables. Only list those parameters that are collected and reported, do not list field parameters. See Table 2 in the "Nutrient and Chlorophyll Monitoring Program and Database Design" SOP version 1.8 (March 2017) document for a full list of directly measured and computed variables. Do not include field parameters in this section.]

a) Parameters measured directly

Nitrogen species: NH4F, NO2F, NO23F

Phosphorus species: PO4F

Other: CHLA_N, PHEA, SiO4F

b) Calculated parameters

NO3F NO23F-NO2F DIN NO23F+NH4F

12) Limits of detection -

[Instructions/Remove: This section explains how the laboratory determines the minimum detection limit (MDL). List the method detection limits used and dates they were in use. You may copy this data from the MDL sheet created in the NutrientQAQC macro. You must also include the date that each MDL was revisited by the lab for appropriateness (this should be done at least annually).]

[Example, update for your laboratory]: 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. These values are reviewed and revised periodically.

<u>Parameter</u>	Start Date	<u>End Date</u>	MDL
<u>CHLA N</u>	01/01/2022	12/31/2022	<u>0.50</u>
<u>NH4F</u>	01/01/2022	12/31/2022	0.0062
NO23F	01/01/2022	12/31/2022	0.0055
NO2F	01/01/2022	12/31/2022	<u>0.0016</u>
PO4F	01/01/2022	12/31/2022	0.0029

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13) Laboratory methods -

[Instructions/Remove: This section lists the laboratory and reference method, the method reference, a brief description of method and a brief description of the sample preservation method used for each parameter that is directly determined.]

a) Parameter: NH4F

Method Reference: Standard Methods 4500-NH₃ H. U.S. EPA 1983. USEPA-600/4-79-020. Method 350.1.

Method Descriptor: Samples were filtered with a 0.45 µm membrane filter and subjected to hypochlorite, which in the presence of phenol, catalytic amounts of nitroprusside and excess hypochlorite, yields indophenol blue, which measured at 630 nm is proportional to the original ammonia concentration.

Preservation Method: Samples are filtered and stored frozen (-20 degC).

Holding Time: 28 days

b) Parameter: NO23F

Method Reference: Standard Methods 4500-NO₃ F.

U.S. EPA 1974. Method 353.2.

Method Descriptor: Samples were filtered with 0.45 um polycarbonate filters. Filtered sample is subjected to cadmium reduction column to reduce nitrate to nitrite. The sample nitrite is then determined by diatizing with sulfanilamide and coupling with N-(1-napthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured at 520 nm and is proportional to the original nitrate + nitrite concentration. The NO2F concentration (below) is subtracted from this result to give NO3F.

Preservation Method: Samples are filtered and stored frozen (-20 degC).

Holding Time: 28 days

c) Parameter: NO2F

Method Reference: Standard Methods 4500-NO₃ F. U.S. EPA 1974. Method 353.2.

Method Descriptor: Samples were filtered with 0.45 um polycarbonate filters. Nitrite in a filtered sample is measured by closing off the cadmium reduction column so that the nitrate is not converted and the sample follows through the same chemistry as with NO3F to yield the original nitrite concentration.

Preservation Method: Samples are filtered and stored frozen (-20 degC).

Holding Time: 28 days

d) Parameter: NO3F

Method Reference: Standard Methods 4500-NO₃ F. U.S. EPA 1974. Method 353.2.

Method Descriptor: Nitrate is calculated from NO23F minus NO2F results. Preservation Method: Samples filtered and stored frozen (-20 degC). Holding Time: Nitrate is calculated from NO23F minus NO2F results.

e) Parameter: DIN

Method: DIN is calculated by adding the NH4F and NO23F results together.

f) Parameter: PO4F

Method Reference: Standard Methods 4500-P E. U.S. EPA 1978. Method 365.1.

Method Descriptor: Samples were filtered with 0.45 um polycarbonate filters. Filtered sample is subjected to ammonium molybdate and antimony potassium tartrate under acidic conditions to form a yellow complex. This complex is reduced with ascorbic acid to form a blue complex, which absorbs light at 880 nm. The absorbance is proportional to the concentration of orthophosphate in the sample.

Preservation Method: Samples are filtered and stored frozen (-20 degC).

Holding Time: 28 days

g) Parameter: CHLA N

Method Reference: EPA 445 REV 1.2

Method Descriptor: Suspended sediment and other material in a water sample is concentrated onto a 47 mm GF/F filter under low vacuum. The sample is stored in a petri dish wrapped in aluminum foil in an airtight plastic bag kept on ice while in the field. The samples are then kept frozen and in the dark until analysis. The acetone extraction method is used to extract the chlorophyll over 2-24 hours and a fluorometer is used to obtain readings, which are calculated into a final result. Preservation Method: Filters are stored frozen (-20 degC).

Holding Time: 28 days

a) Parameter: NH4F

VIMS Laboratory Method: 126

EPA or other Reference Method: 170.1

Method Reference: US.EP.A 1983. USEP.A 600/4-79-020. Method 170.1

Method Descriptor: Filtered sample subjected to hypochlorite phenol...

Preservation Method: Samples filtered and stored at 4 °C up to 24 hours.

b) Parameter: NO2F

VIMS Laboratory Method: 142

EPA or other Reference Method: 167.1

Method Reference: US.EP.A 1983. USEP.A 600/4-79-020. Method 167.1

Method Descriptor: Filtered sample subjected to cadmium reduction column...

Preservation Method: Samples filtered and stored frozen at -20 °C up to 14 days.

14) Field and Laboratory QAQC programs -

[Instructions/Remove: This section describes field variability, laboratory variability, the use of interorganizational splits, sample spikes, standards, and cross calibration exercises. Include any information on QAQC checks performed by your lab.]

a) Precision

- i) Field variability Field replicates are successive grab samples. Duplicate grabs are collected.
 Samples are filtered and placed on ice before the next sample is grabbed (usually about 10 minutes between grabs).
- ii) Laboratory variability All samples are analyzed in duplicates.
- iii) Inter-organizational splits Samples were analyzed by one lab.

Field variability – List the specific number (100%) of field replicates; describe how replicates are collected: true field replicates are successive grab samples, replicates split from a single field sample are considered laboratory replicates/splits.

- Laboratory variability List specific number (10%) of laboratory replicates.
- <u>iii)vi)</u> Inter-organizational splits Specify if samples were split and analyzed by two different labs.

b) Accuracy

- i) The laboratory is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the continued analysis of laboratory reagent blanks, field duplicates (where applicable) and quality control samples as a continuing check on performance. The laboratory will maintain performance records that define the quality of the data generated.
- ii) Initial Daily Calibration Correlation coefficient (r): The correlation coefficient must be 0.995 or better for the calibration curve to be used. Calibration standards should bracket the range of samples analyzed.
- iii) Laboratory Control Sample (LCS) Certified reference material The laboratory must analyze an ammonia certified LCS to verify the accuracy of the initial calibration. Alternatively, a material from a second-source or lot that is traceable to a national standard may be used.
- iv) A CCV standard and blank are analyzed every 10 samples to assess drift throughout the run. If the CCV exceeds 10% of the true value, then a new calibration must be performed and the previous 10 samples reanalyzed. If the blank exceeds the lowest standard, then a new calibration must be performed and the previous 10 sample reanalyzed.
- v) Duplicates and samples spikes, must be performed for every 10 samples analyzed. Duplicate reproducibility should be < 20% with the exception of samples which fall at or below the MDL or in the low end of the calibration curve at the lowest standard. In this instance, a subsequent sample falling in the proper range validates the run. Sample spikes should recover > 80% of the spike added. If the sample does not recover the appropriate spike, the sample and spike should be repeated. If it again does not met the QC requirements, the sample may be determined to have a matrix interference if all other sample spikes in the run have met the

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required QC acceptance limits. This sample will be reported to the client as having a matrix interference.

- vi) A back calculation of calibration standards must be performed. The calibration standard must meet +/-10% of the true value criteria for each point except for standards falling at or below the PQL. Standards falling at or below the PQL must recover at +/- 30% of the true value of the calibration standard.
- vii) A QC sample spiked a 1-2 times the MRL must be analyzed at least quarterly. The MRL should not exceed +/-20% of the back calculated standard.

b)

- i) Sample spikes List the % recovery of field and laboratory samples recovery should be 100% under ideal conditions) - cannot be done on samples analyzed directly from filters
- ii) Standard reference material analysis This will result from samples sent out from EPA to each lab.
- iii) Cross calibration exercises CBNERRVA participates in cross calibration exercises. Cross calibration exercises include the Chesapeake Bay Quarterly Split Sam Program and the US EPA Method Validation Studies.

15) QAQC flag definitions -

Hastructions/Remove: This section details the primary and secondary QAQC flag definitions and requires no additional information. Include the following excerpt.

QAQC flags provide documentation of the data and are applied to individual data points by insertion into the parameter's associated flag column (header preceded by an F_). QAQC flags are applied to the nutrient data during secondary QAQC to indicate data that are out of sensor range low (-4), rejected due to QAQC checks (-3), missing (-2), optional and were not collected (-1), suspect (1), and that have been corrected (5). All remaining data are flagged as having passed initial QAQC checks (0) when the data are uploaded and assimilated into the CDMO ODIS as provisional plus data. The historical data flag (4) is used to indicate data that were submitted to the CDMO prior to the initiation of secondary QAQC flags and codes (and the use of the automated primary QAQC system for WQ and MET data). This flag is only present in historical data that are exported from the CDMO ODIS.

- -4 Outside Low Sensor Range
- -3 Data Rejected due to QAQC
- -2 Missing Data
- -1 Optional SWMP Supported Parameter
- 0 Data Passed Initial QAQC Checks
- 1 Suspect Data
- 4 Historical Data: Pre-Auto QAQC
- 5 Corrected Data

16) QAQC code definitions -

[Instructions/Remove: This section details the secondary QAQC Code definitions used in combination with the flags above and requires no additional information. Include the following excerpt.

QAQC codes are used in conjunction with QAQC flags to provide further documentation of the data and are also applied by insertion into the associated flag column. There are three (3) different code categories, general, sensor, and comment. General errors document general problems with the sample or sample collection, sensor errors document common sensor or parameter specific problems, and comment codes are used to further document conditions or a problem with the data. Only one general or sensor error and one comment code can be Formatted: No bullets or numbering

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applied to a particular data point. However, a record flag column (F_Record) in the nutrient data allows multiple comment codes to be applied to the entire data record.

General errors GCM Calculated value could not be determined due to missing data GCR Calculated value could not be determined due to rejected data GDM Data missing or sample never collected GQD Data rejected due to QA/QC checks GQS Data suspect due to QA/QC checks See metadata GSM Sensor errors SBL Value below minimum limit of method detection Calculated value could not be determined due to a below MDL component **SCB** SCC Calculation with this component resulted in a negative value SNV Calculated value is negative SRD Replicate values differ substantially SUL Value above upper limit of method detection Parameter Comments CAB Algal bloom CDR Sample diluted and rerun CHB Sample held beyond specified holding time CIP Ice present in sample vicinity CIF Flotsam present in sample vicinity CLE Sample collected later/earlier than scheduled **CRE** Significant rain event **CSM** See metadata CUS Lab analysis from unpreserved sample Record comments CAB Algal bloom CHB Sample held beyond specified holding time CIP Ice present in sample vicinity CIF Flotsam present in sample vicinity CLE Sample collected later/earlier than scheduled CRE Significant rain event **CSM** See metadata CUS Lab analysis from unpreserved sample Cloud cover clear (0-10%) CCL CSP scattered to partly cloudy (10-50%) CPB partly to broken (50-90%) COC overcast (>90%) CFY foggy CHY hazy CCC cloud (no percentage) Precipitation PNP none PDR drizzle

PLR

PHR

PSQ

PFQ

light rain

squally

heavy rain

frozen precipitation (sleet/snow/freezing rain)

PSR mixed rain and snow Tide stage TSE ebb tide TSF flood tide TSH high tide TSL low tide Wave height WH0 0 to < 0.1 meters WH1 0.1 to 0.3 meters WH2 0.3 to 0.6 meters WH3 0.6 to > 1.0 metersWH4 1.0 to 1.3 meters WH5 1.3 or greater meters Wind direction from the north Ν NNE from the north northeast NE from the northeast ENE from the east northeast E from the east ESE from the east southeast SE from the southeast SSE from the south southeast S from the south SSW from the south southwest SWfrom the southwest WSW from the west southwest W from the west WNW from the west northwest NWfrom the northwest NNWfrom the north northwest Wind speed WS0 0 to 1 knot WS1 > 1 to 10 knots WS2 > 10 to 20 knots WS3 > 20 to 30 knots WS4 > 30 to 40 knots WS5 > 40 knots

17) Other remarks/notes -

[Instructions/Remove: Use this section for further documentation of the data set. Include any additional notes regarding the data set in general, circumstances not covered by the flags and comment codes, or specific data that were coded with the CSM "See Metadata" comment code. Any data coded CSM must have a corresponding statement in this section. You may include the metadata worksheets here if so desired. You may also include information on major storms or precipitation events that could have affected the data recorded. You must include a table (not an image of a table) detailing sample/parameter collection and processing dates. Include the following excerpt.]

- Analysis of TSS/TFS/TVS was discontinued after March of 2022. The reason for this is unknown.

No nutrient data (grabs or diel samples) were taken in May 2022 due to staff shortages.

- No diel samples were taken in July of August 2022 due to instrument (ISCO 6712) malfunction.

No times were recorded for the diel samples taken in June, but dates were recorded.

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Three different individuals took nutrient samples throughout 2022. Thompson Rose (SWMP Manager) took samples from January - March. Rachel Guy (Research Coordinator) took samples from April - July. Ivanna Knox (SWMP Manager) took samples from August - December.

Data may be missing due to problems with sample collection or processing. Laboratories in the NERR System submit data that are censored at a lower detection rate limit, called the Method Detection Limit or MDL. MDLs for specific parameters are listed in the Laboratory Methods and Detection Limits Section (Section II, Part 12) of this document. Concentrations that are less than this limit are censored with the use of a QAQC flag and code, and the reported value is the method detection limit itself rather than a measured value. For example, if the measured concentration of NO23F was 0.0005 mg/l as N (MDL=0.0008), the reported value would be 0.0008 and would be flagged as out of sensor range low (-4) and coded SBL. In addition, if any of the components used to calculate a variable are below the MDL, the calculated variable is removed and flagged/coded -4 SCB. If a calculated value is negative, it is rejected and all measured components are marked suspect. If additional information on MDL's or missing, suspect, or rejected data is needed, contact the Research Coordinator at the reserve submitting the data.

Note: The way below MDL values are handled in the NERRS SWMP dataset was changed in November of 2011. Previously, below MDL data from 2007-2010 were also flagged/coded, but either reported as the measured value or a blank cell. Any 2007-2011 nutrient/pigment data downloaded from the CDMO prior to November of 2011 will reflect this difference.

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[Example explanation, update for your sample storage protocols] Sample hold times for 2022: Samples are held at -20°C. NERRS SOP allows nutrient samples to be held for up to 28 days (CHLA for 30) at -20°C, plus allows for up to 5 days for collecting, processing, and shipping samples. Samples held beyond that time period are flagged suspect <1>and coded (CHB). If measured values were below MDL, this resulted in <-4> [SBL] (CHB) flagging/coding.

[Example explanation 2, update for your sampling protocols] Sample hold times for 2022: NERRS SOP allows nutrient samples to be held for up to 24 hours if held at 4°C with no preservation, for NH4F and NO23F up to 28 days if acidified and held at 4°C, and up to 28 days (CHLA for 30 days) if held at -20°C. Tier II parameters, with a few exceptions, are subject to the same sample hold times. In all cases, up to an additional 5 days is allowed for collecting, processing, and shipping samples. Samples held beyond that time period are flagged suspect and coded CHB in the data set.

[Example table, format however makes sense for your reserve]

A						[I	Formatted: Font: Bold, Underline
			Date	of Analysis			Formatted: Font: Bold, Underline
<u>Sample</u>	PO4F	NH4F	NO2F	NO23F	CHLA N	TSS/TFS/TV	<u>'S</u>
Descriptor							
1/11/2022, grab	2/2/2022	2/2/2022	2/2/2022	2/2/2022	1/18/2022	1/13/2022	
and diel samples							
2/10/2022, grab	2/24/2022	2/24/2022	2/24/2022	2/24/2022	2/17/2022	2/17/2022	
and diel samples							
3/25/2022, grab	4/13/2022	4/13/2022	4/13/2022	4/13/2022	4/21/2022	3/29/2022	
and diel samples							
4/25/2022, grab	5/24/2022	5/24/2022	5/24/2022	5/24/2022	5/11/2022	◆ <u>-</u> 1	Formatted: Centered
and diel samples							
MISSING MAY	Ξ	=	<u>=</u>	=	Ξ	4	Formatted: Centered
<u>SAMPLES</u>							
6/23/2022, grab	7/14/2022	7/14/2022	7/14/2022	7/14/2022	7/6/2022	◆ <u>-</u> 1	Formatted: Centered
and diel samples							
7/12/2022, grab	8/3/2022	8/3/2022	8/3/2022	8/3/2022	7/20/2022	4 <u>-</u>	Formatted: Centered
and diel samples							
8/19/2022, grab	9/6/2022	9/6/2022	9/6/2022	9/6/2022	9/1/2022	4 <u>-</u>	Formatted: Centered
and diel samples							
9/20/2022, grab	10/18/2022	10/18/2022	10/18/2022	10/18/2022	10/17/2022	◆ <u>-</u>	Formatted: Centered
and diel samples							
10/20/2022,	11/15/2022	11/15/2022	11/15/2022	11/15/2022	11/10/2022	4	Formatted: Centered
grab and diel							
samples							
11/23/2022,	12/13/2022	12/13/2022	12/13/2022	12/13/2022	12/15/2022	4 <u>-</u>	Formatted: Centered
grab and diel							
samples							
12/22/2022,	1/19/2022	1/19/2022	1/19/2022	1/19/2022	1/9/2022	4	Formatted: Centered
grab and diel							
samples							

Data of analysis		
	Data of analysis	

Sample Descriptor	PO4F	NH4F	NO2F	NO23F	CHLA_N, PHEA	◆ SiO4F Forma	tted 1
1/4/2022, all grabs	1/13/2022	1/13/2022	1/13/2022	1/13/2022	1/12/2022	1/21/2022	
2/29/2022, all grabs	3/24/2022	3/24/2022	3/24/2022	3/24/2022	3/21/2022	4/1/2022	
2/29-3/1/2022, all diels	3/24/2022	3/24/2022	3/24/2022	3/24/2022	3/21/2022	4/1/2022	
3/28/2022, all grabs	4/22/2022	4/22/2022	4/22/2022	4/22/2022	5/10/2022*	5/10/2022*	
3/30-3/31/2022, all diels	4/22/2022	4/22/2022	4/22/2022	4/22/2022	4/18/2022	5/4/2022	
4/25/2022, all grabs	5/20/2022	5/20/2022	5/20/2022	5/20/2022	5/11/2022	5/23/2022	
4/25-4/26/2022, all diels	5/20/2022	5/20/2022	5/20/2022	5/20/2022	5/17/2022	5/23/2022	
5/2/2022, all grabs	5/20/2022	5/20/2022	5/20/2022	5/20/2022	5/24/2022	5/23/2022	
5/16-17/2022, all diels	6/8/2022	6/8/2022	6/8/2022	6/8/2022	6/1/2022	6/10/2022	
	-	_	_	-	-	-	

 $[\]ensuremath{^*} \text{sample}$ held longer than allowed by NERRS protocols