

Great Bay Estuary (GRB) NERR Nutrient Metadata

April 2013 through December 2013

Latest Update: ~~xxxx~~May 15, 2014

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

Months and year the documentation covers

Latest Update: Date that the last edits were made

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I. Data Set and Research Descriptors

1) 1) Principal Investigator(s) and Contact Persons

a) Principal Investigator

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Principal investigator(s) and contact persons — 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.

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2) ~~2)~~ Research Objectives

Nutrient monitoring in GBNERR has been conducted since the late 1980s with the basic goal of developing and maintaining temporally intensive long-term datasets of physico-chemical parameters of water quality at locations that are representative of the Great Bay estuarine system. The Great Bay site is relatively unimpacted, while the three tidal river sites (Lamprey, Oyster and Squamscott) have large drainage basins and are impacted by both point (wastewater treatment plants) and nonpoint sources of pollution. In addition to establishing a baseline of water quality and understanding the spatial and temporal variability of important indicators of estuarine water quality, the data are used by researchers in the analysis of physical and biological processes and by decision makers in the management of the estuary and watershed.

a) Monthly Grab Sampling Program

Monthly grab samples are collected to quantify the horizontal spatial variability of important nutrients in the water column at sites located in the upper and lower estuary representative of the local salinity and habitat gradients.

b) Diel Sampling Program

Once per month, samples are collected over a complete lunar day to quantify the temporal variability of important nutrients in the water column as a function of tidal dynamics.

Research objectives — Describe briefly the nature of the monitoring program resulting in this data set (monitoring along land use, vertical, salinity or habitat gradients).

Monthly Grab Sampling Program

Diel Sampling Program (mention if samples were taken over a lunar day)

3) Research Methods

a) Monthly Grab Sampling Program

Monthly grab samples are collected at the Great Bay buoy, Squamscott River, Oyster River and Lamprey River locations. All grab samples from these stations were collected on ebbing tides within 3 hours of low tide in the same 24-hour period. Under normal conditions, most samples are taken by boat except Oyster River which is almost always sampled from a floating dock and

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Lamprey River which is occasionally sampled from a floating dock. Historically, two replicate samples were collected at each station at a depth of approximately 0.5 m from the surface. Beginning with the June 2009 sampling, a single sample is collected at three of the four stations and a triplicate sampling is performed at the fourth station at least every other month. The station replicated is chosen randomly. Each bottle is opened individually, rinsed three times with estuarine water then filled facing into the current, if any current pattern is detected. For replicates, subsequent samples are collected immediately following the first. All samples are collected in polyethylene sample bottles that were previously acid washed (10% HCl), rinsed (3x) with deionized water and dried. Filled bottles are capped and stored in a dark cooler for the remainder of the sampling cruise. Upon arrival to the laboratory, samples are processed immediately.

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Field parameter data including salinity, temperature, dissolved oxygen and dissolved oxygen percent saturation are collected using a YSI Model 85 Handheld Dissolved Oxygen and Conductivity Instrument. The meter is operated and calibrated according to manufacturer instructions. Measurements are made at grab sample depth (0.5 m) immediately following grab sample collection.

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Light attenuation readings in the water column are taken using a LI-COR LI-193 Spherical Quantum Sensor while incident solar irradiance is measured with a LI-COR LI-190 Quantum Sensor. Data from both sensors are logged with a LI-COR LI-1400 Datalogger. Care is taken to conduct individual casts during times of consistent solar irradiance. Measurements are made at least every 0.25 m in the upper 2 m then less frequently deeper. Where possible, eight or more readings are taken per cast. The light attenuation coefficient for PAR (K_d) is obtained by computing the linear regression of sample depth vs. $\ln$ (PAR) then taking the absolute value of the inverse of the slope of the regression. Values for K_d are considered robust if the r^2 of the regression is >0.95 and values failing this test are not included in the dataset. It is usually impossible to do the light attenuation measurement at the Oyster River site due to shallow water depth.

b) Diel Sampling Program

Once per month a Sigma autosampler is deployed from a fixed wharf near the sonde location in the Lamprey River. Historically, this device automatically collected two 850 ml water samples one minute apart every 2 hours and 30 minutes for 22.5 hours such that 10 time points were sampled, each with a replicate. Beginning with the May 2009 sampling, the sampling scheme was changed to ensure that sampling incorporated a full lunar day while improving temporal resolution. Samples are now collected at 2 hour and 4 minute intervals over the lunar day such that 13 time points are sampled. While not required by SWMP protocols, triplicate samples are usually taken at either the first or final time points and are often collected at both time points. An effort is made to begin sampling at dead low tide although this is not always possible. All samples are pumped into polyethylene sample bottles that were previously acid washed (10% HCL), rinsed (3x) with distilled-deionized water and dried. At the end of the sampling period, the samples are kept in the dark and returned to the laboratory for immediate processing.

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4) Site Location and Character

The Great Bay is a macro-tidal drowned river valley estuary located in northern New England. Seven major tributaries contribute runoff to Great Bay draining a total area of roughly 2,400 km². Strong tidal and wind driven currents drive circulation patterns, vertical mixing, and resuspension of sediments, which in turn affect primary productivity. The estuary contains five major habitat types in the form of mud flat, eelgrass meadows, salt marsh, channel bottom, and rocky shore. Four permanent stations are monitored as a part of the water quality and nutrient long-term monitoring programs. They are located in mid Great Bay, and in the Lamprey, Oyster and Squamscott Rivers.

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Site #1 Great Bay (GB)

Location: Central area of Great Bay proper.

Coordinates: 43° 04' 20" N latitude and 70° 52' 10" W longitude.

Salinity range: 5-32 ppt (seasonally); 0-5ppt from high to low tide.

Temperature range: -1°C to 24°C (seasonally); 0-3°C (from high to low tide)

Depth: 6.5 meters at MLW

Tidal height: 2.7 meters

Bottom type: Mud and rock channel bottom

Tidal velocity: maximum 50 cm/sec

Watersheds: Squamscott, Lamprey and Winnicut Rivers plus smaller streams. High tide influence from Little Bay and associated rivers

Pollutant influence: clean reference site

Data sonde deployment (GRB WQ monitoring data set): The sonde was mounted on a communications buoy at 1 meter below the surface. The sonde data was telemetered to the lab base station via spread spectrum radio. As a back-up measure, the sonde continued to log in unattended mode to avoid potential data loss due to communications failure.

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Site #2 Squamscott River (SQ)

Location: Mid channel of the Squamscott River at the Boston and Maine Railroad bridge, Stratham, NH.

Coordinates: 43° 02' 30" N latitude and 70° 55' 20" W longitude

Salinity range: 0-30ppt (seasonally); 5-20 ppt from high to low tide

Temperature range: -1°C to 27°C (seasonally); difference of 0-5°C from high to low tide

Depth: 3.5 meters at MLW

Tidal height of 2.7 m

Bottom type: Mud/oyster channel bottom

Tidal velocity: maximum 50 cm/sec

Watersheds: Exeter River, adjacent marshes

Pollutant influence: Urban storm water, agriculture, two municipal wastewater treatment plants, residential septic systems

Data sonde deployment (GRB WQ monitoring data set): The sonde was deployed in a perforated PVC cylinder attached to a piling at a fixed 0.5m height above the bottom and

connected to a Nexsens iSIC, equipped with a cellular modem, transmitting to the lab base station. The sonde logged in unattended mode for data redundancy.

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Site #3 Lamprey River (LR)

Location: West bank of the tidal portion of the Lamprey River, approximately 300 m downstream of the dam at Route 108 in Newmarket, NH

Coordinates: 43° 04' 48" N latitude and 70° 56' 04" W longitude

Salinity range: 0 to 27 (seasonally); difference of up to 15 ppt between high and low tides

Temperature range: -1°C to 27°C (seasonally); difference of 0-5°C between high and low tides

Depth: 3.5 m

Tidal height 2.7 m

Bottom type: mud/rock

Tidal velocity: maximum 40 cm/sec

Watershed: Lamprey River

Pollutant influence: Urban stormwater, adjacent marina, upstream and downstream wastewater treatment plants, upstream agriculture

Data sonde deployment (GRB WQ monitoring data set): The sonde was deployed in a perforated PVC cylinder attached to a piling at a fixed height of 0.5 m above the bottom. The sonde was telemetered via cellular modem using a Nexsens iSIC. The sonde logged in unattended mode to ensure data acquisition at all times.

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Site #4 Oyster River (OR)

Location: in the center channel of the tidal portion of the Oyster River, approximately 300 m downstream of the head of tide dam adjacent to Jackson's Landing in Durham, NH.

Coordinates: 43° 08' 2" N latitude and 70° 54' 40" W longitude (43.134 N, 70.911 W in Decimal Degrees)

Salinity range: 0 to 32 (seasonally); difference of up to 15 ppt between high and low tides

Temperature range: -1°C to 27°C (seasonally); difference of up to 5°C between high and low tides

Depth: 0.3 m at mlw, 3 m at highest high tides

Tidal height 2.7 m (maximum)

Bottom type: mud

Tidal velocity: maximum 40 cm/sec

Watershed: Oyster River

Pollutant influence: urban stormwater, mooring field and crew dock, downstream wastewater treatment plant, upstream agriculture, residential on-site sewage disposal

Data sonde deployment (GRB WQ monitoring data set): Deployed vertically in a PVC tube attached to the stem of a mushroom anchor, approx. 0.5 meters above bottom. The Oyster River sonde was telemetered by a Sutron GOES satellite transmitter and logged simultaneously in unattended mode.

Research methods—Detail the specifics of sample collection, collection intervals, sample processing, QA/QC of the equipment and analyzers.

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Monthly Grab Sampling Program

Diel Sampling Program

4) Site location and character—Describe your NERR site in general and the sampling sites associated with each YSI data logger / nutrient collection. Include the following 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:

- _____ a) latitude and longitude
- _____ b) tidal range
- _____ c) 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

5) Coded Variable Definitions

GRB = Great Bay Estuary NERR

GB = Great Bay Site

LR = Lamprey River Site

SQ = Squamscott River Railroad Trestle Site

OR = Oyster River Site

NUT = Nutrient Sampling Program

1 = Monthly Grab Sample Program

2 = Diel Grab Sample Program

For example, as sample code of 'GRBLRNUT' indicates that the sample was collect in the Great Bay NERR at the Lamprey River Site and is being reported as part of the Nutrient Sampling Program Data Report. A Monitoring Program Code of '2' indicates that the sample was collected as part of the Diel Grab Sample Program.

Coded variable definitions— Explain the station code names and monitoring program codes. For example:

- _____ cbvtcnut = Chesapeake Bay Virginia Taskinas Creek nutrients
- _____ monthly grab sample program = 1
- _____ diel grab sample program = 2

6) Data Collection Period

Sampling in the Great Bay Estuary is typically limited to the months of April through November as a result of ice formation during the winter months. We were not able to perform a diel sampling in December 2013 however we were able to collect grab samples at three of the four stations. For 2013, the first diel collection was on April 29, 2013 and the first grab samples were

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collected on April 22, 2013. The final grab samples were collected on December 10, 2013. The final diel cycle sampling was completed on November 21, 2013.

Grab Sample Collection:

Station: grbgb Sample Date and Time

4/22/2013 15:20
4/22/2013 15:21
4/22/2013 15:22
5/22/2013 14:22
6/19/2013 13:45
7/9/2013 8:00
7/9/2013 8:01
7/9/2013 8:02
8/14/2013 12:20
9/10/2013 9:10
9/10/2013 9:11
9/10/2013 9:12
10/8/2013 9:10
11/14/2013 16:47
12/10/2013 12:01
12/10/2013 12:02
12/10/2013 12:03

Station: grbor Sample Date and Time

4/22/2013 14:10
5/22/2013 15:00
6/19/2013 14:30
7/9/2013 7:10
8/14/2013 10:10
8/14/2013 10:11
8/14/2013 10:12
9/10/2013 9:20
10/8/2013 8:10
11/14/2013 13:35

Station: grbsq Sample Date and Time

4/22/2013 15:00
5/22/2013 14:10
6/19/2013 13:20
6/19/2013 13:21
6/19/2013 13:22

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Station: grblr Sample Date and Time

Diel Sample Collection:

7) Associated Researchers and Projects

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The SWMP nutrient data set is one of several water quality monitoring programs in the Great Bay Estuary that include both physical and chemical properties. More information including over ten years of additional nutrient data can be found at <http://www.ciceet.unh.edu/>. A spatially explicit non-exhaustive list and description of additional programs can be found at <http://www.gulfofmaine.org/esip/map/>.

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8) Distribution

NOAA/ERD retains the right to analyze, synthesize and publish summaries of the NERRS System-wide Monitoring Program 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 this 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 Estuarine Reserves Division, Office of Ocean and Coastal Resource Management, National Ocean Service, National Oceanic and Atmospheric Administration. 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.

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 <http://cdmo.baruch.sc.edu/>. Data are available in text tab-delimited format.

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Data collection period—List the date and time each sample was collected (include start and end times). Specify the date that SWMP nutrient monitoring first began for each monitoring site.

7) Associated researchers and projects (link to other products or programs)—Describe briefly other research (data collection) that correlates or enhances the nutrient data. At a minimum, mention the SWMP-MET and WQ datasets.

8) Distribution—This section will address data ownership and data liability by including the following excerpt from the Ocean and Coastal Resource Management Data Dissemination Policy for the NERRS System-wide Monitoring Program in the metadata.

NOAA/ERD retains the right to analyze, synthesize and publish summaries of the NERRS System-wide Monitoring Program 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 this NOAA/OCRM supported research that are produced

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for publication in open literature, including refereed scientific journals, will acknowledge that the research was conducted under an award from the Estuarine Reserves Division, Office of Ocean and Coastal Resource Management, National Ocean Service, National Oceanic and Atmospheric Administration. 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.

Also include the following excerpt in the metadata which will 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 <http://cdmo.baruch.sc.edu/>. Data are available in text tab delimited format.

II. Physical Structure Descriptors

9) Entry Verification

All field and lab data were collected following protocols described in the project QAPP. Data were entered into an Excel Spreadsheet, verified by UNH laboratory technicians and/or the Principal Investigator.

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.

10) Parameter Titles and Variable Names by Data Category

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The following is a list of water-quality and environmental parameters included in the GRB SWMP Nutrient Database. Required NOAA/NERRS System-wide Monitoring Program water quality parameters are denoted by an asterisks “*”.

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Data Category	Parameter	Variable Name	Units of Measure
a) Phosphorus:			
	*Orthophosphate, Filtered	PO4F	mg/L as P
b) Nitrogen:			
	*Nitrite + Nitrate, Filtered	NO23F	mg/L as N
	*Ammonium, Filtered	NH4F	mg/L as N
	Dissolved Inorganic Nitrogen	DIN	mg/L as N
	Dissolved Organic Nitrogen	DON	mg/L as N
	Total Dissolved Nitrogen	TDN	mg/L as N
	Particulate Organic Nitrogen	PON	mg/L as N
c) Plant Pigments:			
	*Chlorophyll a	CHLA	µg/L
	Phaeophytin	PHEA	µg/L
d) Other Lab Parameters:			
	Dissolved Organic Carbon	DOC	mg/L as C
	Particulate Organic Carbon	POC	mg/L as C
	Total Suspended Solids	TSS	mg/L
e) Field Parameters:			
	Water Temperature	-WTEM N	°C
	-Salinity	-SALT N	ppt
	-Dissolved Oxygen	-DO N	mg/L
	-Dissolved Oxygen Saturation	-DO S N	%
	-Light Extinction Coefficient	-Kd N	m ⁻¹

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Notes:

1. Time is coded based on a 2400 hour clock and is referenced to Standard Time.
2. Reserves have the option of measuring NO₂ and NO₃, or they may substitute NO₂₃ for individual analyses if they can show that NO₂ is a minor component relative to NO₃. NO₂ has been shown to be a minor component relative to NO₃ in Great Bay and so, beginning in 2009, Great Bay no longer reports NO₂ and NO₃ separately.

11) Measured and Calculated Laboratory Parameters

The following parameters are reported in the 2011 Nutrient Data Report for GRB

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a) Variables Measured Directly

Nitrogen species: NH₄F, NO₂3F, TDN

Phosphorus species: PO₄F

Other: TSS, CHLA, PHEA, ~~DOC~~, POC, PON

b) Computed Variables

DIN: NO₂3F+NH₄F

DON: TDN-DIN

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c) Measured Environmental Variables

Water Temperature, Salinity, Dissolved Oxygen, Dissolved Oxygen Saturation, Light Attenuation, Cloud Cover, Precipitation, Tidal Stage, Wave Height, Wind Direction, Wind Speed.

12) Limits of Detection

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The following Method Detection Limits (MDL), the lowest concentration of a parameter that an analytical procedure can reliably detect, have been established for the UNH laboratory that performed analyses in 2013 (Table 1). Concentrations below the MDL are reported as blank cells in the database and are flagged accordingly. Samples that fall above the maximum range of the chemistry are diluted so that the analysis falls within the analytical range of the chemistry and these values are multiplied by the dilution factor (e.g. 10 for a 1:10 dilution) and reported in the database. MDL values are reviewed and revised periodically.

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Table 1. University of New Hampshire Water Quality Analysis Laboratory Method Detection Limits (MDL) for measured water quality parameters.

Parameter	Start Date	End Date	MDL
CHLA N	01/01/13	13/31/13	0.1296
NH4F	01/01/13	13/31/13	0.005
NO23F	01/01/13	13/31/13	0.005
PHEA	01/01/13	13/31/13	0.1296
PO4F	01/01/13	13/31/13	0.005
TDN	01/01/13	13/31/13	0.0395
TSS	01/01/13	13/31/13	1
DOC	01/01/13	13/31/13	0.05
POC	01/01/13	13/31/13	0.02
PON	01/01/13	13/31/13	0.02

13) Laboratory Methods

The University of New Hampshire Water Quality Analysis Laboratory was responsible for the processing of the 2013 GRB nutrient data; methods for each parameter will be presented below.

a) Parameter: NH4F

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i) Method Reference: Westco Scientific Instruments, SmartChem Discrete Analyzer. SmartChem Method #210-200B. Method is based on USEPA 350.1. Spectrophotometric and Kinetics Investigation of the Bertholt Reaction for the Determination of Ammonia, Analytical Chemistry, 1977, Vol. 49, #3, P.464-469.

ii) Method Descriptor: This method is based on the Bertholt reaction. Ammonia reacts in alkaline solution with hypochlorite to form monochloramine which, in the presence of phenol, catalytic amounts of nitroprusside (nitroferricyanide) and excess hypochlorite, gives indophenol blue. The formation of monochloramine requires a pH between 8 and 11.5. At higher pH, ammonia may begin to oxidize to nitrate. At pH greater than 9.6, some precipitation of calcium and magnesium as hydroxides and carbonates occurs in seawater, but these ions may be held in solution by complexing them with EDTA. The indophenol blue measured at 630 nm is proportional to the original ammonia concentration.

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

b) Parameter: NO23F

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i) Method Reference: Westco Scientific Instruments, SmartChem Discrete Analyzer. SmartChem Method #375-100D. Method is based on USEPA 353.3. Armstrong, F.A., Stearns, C.R. and Strickland, J.D.H. (1967) The measurement of upwelling and subsequent biological processes by means of the Technicon AutoAnalyzer and associated equipment. *Deep Sea Research* 14:381-389.

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ii) Method Descriptor: Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotization with sulfanilamide under acidic conditions to form a diazonium ion. The resulting diazonium ion is coupled with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting pink dye absorbs at 520 nm. The procedure is the same for the nitrite analysis less the cadmium column. Nitrate concentrations are obtained by subtracting nitrite values, which have been previously analyzed, from the nitrite + nitrate values.

Though the method is written for seawater and brackish water, it is also applicable to non-saline sample matrixes. The method is calibrated using standards prepared in deionized water. Once calibrated, samples of varying salinities (0 to 35 ppt) may be analyzed. The determination of background absorbance is necessary only for samples which have color absorbing at 540 nm. The salt effect is less than 2%.

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

c) Parameter: PO4F

University of New Hampshire

i) Method Reference: Westco Scientific Instruments, SmartChem Discrete Analyzer. SmartChem Method #410-200E. Method is based on USEPA 365.2. Bernhardt, H. and Wilhelms, A. (1967) The continuous determination of low level iron, soluble phosphate, and total phosphate with the AutoAnalyzer. *Technicon Symp.* 1:386.

ii) Method Descriptor: Ammonium molybdate and antimony potassium tartrate reacts in an acid medium with phosphate to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color produced is proportional to the phosphate concentration in the sample. Though there is a density difference between seawater and reagent water the bias is less than 2%. Though the method is written for seawater and brackish water it is also applicable to non-saline sample matrixes. The method is calibrated using standards prepared in deionized water. Once calibrated, samples of varying salinities (0 to 35 ppt) may be analyzed. The determination of background absorbance is necessary only for samples, which have color absorbing at 880 nm.

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

d) Parameter: TDN

- i.) Method Reference: Shimadzu Scientific Instruments Inc., TOC-V with TNM-1 Nitrogen Module. High Temperature Catalytic Oxidation with chemiluminescent detection. Merriam, J.L., W.H. McDowell, W.S. Currie, 1996. A high-temperature catalytic oxidation technique for determining total dissolved nitrogen. Soil Science Society of America Journal, 60(4) 1050-1055.
- ii.) Method Descriptor: A precisely measured aliquot of filtered sample is injected and combusted on a catalyst at 720 C. All fixed N is converted to Nitric Oxide (NO) and then coupled with ozone (O₃) producing Nitrogen Dioxide* (NO₂*) which is measured chemiluminescently.
- iii.) Preservation Method: Sample is filtered through a 0.45 um disposable disk filter and stored at -20oC until analyzed.

e) Parameter: DOC

- i.) Method Reference: Shimadzu Scientific Instruments Inc., TOC-V: High Temperature Catalytic Oxidation: USEPA Method 415.1.
- ii.) Method Description: Organic carbon in a sample is converted to carbon dioxide by catalytic combustion or wet chemical oxidations. The carbon dioxide formed is measured directly by an infrared detector. The amount of carbon dioxide is directly proportional to the concentration of carbonaceous material in the sample.
- iii.) Preservation Method: Sample is filtered through a 0.45 um disposable disk filter and stored at -20oC until analyzed.

f) Parameter: POC/PON

- i.) Method Reference: Carbon, Nelson et al, 1996. Total carbon, organic carbon and organic matter. Soil Sci Soc Am ; Nitrogen, Bremner et al., 1996. Nitrogen total. Methods of Soil Analysis.
- ii.) Method Description: An accurately measured amount of particulate matter is combusted at 975C using an elemental analyzer. The combustion products are passed over a copper reduction tube. Carbon dioxide, water vapor, and nitrogen are homogeneously mixed at a known volume, temperature and pressure. The mixture is released to a series of thermal conductivity detectors/traps, measuring in turn by difference, hydrogen (as water vapor), C (as CO₂) and N (as N₂).

iii.) Preservation Method: dried at 60C.

g) Parameter: TSS

University of New Hampshire

- i) Method Reference: JEL SOP 1.06; adapted from Standard Methods for the Examination of Water and Wastewater. 2540 B. pp. 2-55-56.
- ii) Sample water (100-600 ml) is vacuum filtered through a Whatman GF/C pre-weighed 47mm glass microfiber filter. Place filter in drying oven at 80 degrees C for 24 hours for determination of total suspended solids.

h) Parameters CHLA and PHEA

- i) Method Reference: EPA Method 445.0. In Vitro Determination of Chlorophyll a and Pheophytin a in Marine and Freshwater Algae by Fluorescence.

- ii) Sample water (60 – 200 ml) is vacuum filtered through a 25 mm Whatman GF/C glass microfiber filter. The filter is then flash frozen immediately in liquid nitrogen until further processing. Filters are then mixed with 90% Acetone and set into a centrifuge tube to be refrigerated overnight. The following day, samples are vortexed then centrifuged for five minutes. Fluorescence is then determined before and after acid addition using a Turner Aquafluor fluorometer.

GOT HERE 4/19/12, NEED TO GO BACK AND DO SAMPLE DATES/TIMES for DIEL only...

14) QA/QC Programs

a) Precision

- i. Field Variability – GBNERR collects a triplicate at one station at least every two months for grab samples and ~~two~~ usually collects one triplicates per month for diel samples for the determination of water mass variability.
- ii. Laboratory Variability – none
- iii. Inter-organizational splits – none

b) Accuracy

- i. Sample Spikes – blanks
- ii. Standard Reference Material Analysis – see lab protocols
- iii. Cross Calibration Exercises - none

II. Physical Structure Descriptors

9) Entry verification – This section explains how data acquisition, data entry, and data verification (QA/QC) 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

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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 automatically flags/codes values below 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: 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/L 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

Required NOAA/NERRS System-wide Monitoring Program nutrient parameters are denoted by an asterisks "*". 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 1.5 (January 2011) for a full list of available parameters.

Data Category	Parameter	Variable Name	Units of Measure
Phosphorus and Nitrogen:			
	*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	mg/L as N
	Dissolved Inorganic Nitrogen	DIN	mg/L as N
Plant Pigments:			
	*Chlorophyll a	CHLA_N	$\mu\text{g/L}$
	Phaeophytin	PHEA	$\mu\text{g/L}$
Carbon:			
Other Lab Parameters:			
	Silicate, Filtered	SiO4F	mg/L as Si
Microbial:			
Field Parameters:			
	Water Temperature	WTEM_N	$^{\circ}\text{C}$

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—This section lists all measured and calculated variables. Only list those parameters that are collected and reported. See Table 2 in the “Nutrient and Chlorophyll Monitoring Program and Database Design” SOP version 1.5 (January 2011) document for a full list of directly measured and computed variables.

a) **Parameters measured directly**

Nitrogen species: NH₄, NO₂, NO₂₃
Phosphorus species: PO₄F
Other: CHLA, PHEA, SiO₄, WTEM

b) **Calculated parameters**

NO₃ NO₂₃-NO₂
DIN NO₂₃+NH₄

12) Limits of detection—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.

Example: 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.

Parameter	Start Date	End Date	MDL
<u>PO4F</u>	<u>1/1/10</u>	<u>5/31/10</u>	<u>0.0006</u>
<u>PO4F</u>	<u>6/1/10</u>	<u>12/31/10</u>	<u>0.0008</u>
<u>NH4F</u>	<u>1/1/10</u>	<u>12/31/10</u>	<u>0.0015</u>
<u>NO2F</u>	<u>1/1/10</u>	<u>12/31/10</u>	<u>0.0002</u>
<u>NO23F</u>	<u>1/1/10</u>	<u>12/31/10</u>	<u>0.0008</u>
<u>CHLA-N</u>	<u>1/1/10</u>	<u>12/31/10</u>	<u>0.02</u>

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13) Laboratory methods—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**

VIMS Laboratory Method: 126
EPA or other Reference Method: 170.1
Method Reference: US EPA 1983. USEPA 600/4-79-020. Method 170.1
Method Descriptor: Filtered sample subjected to hypochlorite-phenol...

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

~~b) Parameter: **NO3F**~~

~~VIMS Laboratory Method: 142~~

~~EPA or other Reference Method: 167.1~~

~~Method Reference: US EPA 1983. USEPA 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—This section describes field variability, laboratory variability, the use of inter-organizational splits, sample spikes, standards, and cross calibration exercises.~~

~~a) Precision~~

~~i) Field variability—List the specific number (10%) of field replicates; describe how replicates are collected: are field replicates split from a single sample or are they true field replicates (successive grab samples).~~

~~ii) Laboratory variability—List specific number (10%) of laboratory replicates.~~

~~iii) Inter-organizational splits—Specify if samples were split and analyzed by two different labs.~~

~~b) Accuracy~~

~~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 Sample Program and the US EPA Method Validation Studies.~~

15) QAQC flag definitions – This section details the primary and secondary QAQC flag definitions. 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

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- 1 Suspect Data
- 4 Historical Data: Pre-Auto QAQC
- 5 Corrected Data

16) QAQC code definitions – This section details the secondary QAQC Code definitions used in combination with the flags above. ~~Include the following excerpt:~~

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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 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

Sensor errors

SBL	Value below minimum limit of method detection
SCB	Calculated value could not be determined due to a below MDL component
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
<i>Cloud cover</i>	
CCL	clear (0-10%)
CSP	scattered to partly cloudy (10-50%)
CPB	partly to broken (50-90%)
COC	overcast (>90%)
CFY	foggy
CHY	hazy
CCC	cloud (no percentage)
<i>Precipitation</i>	
PNP	none
PDR	drizzle
PLR	light rain
PHR	heavy rain
PSQ	squally
PFQ	frozen precipitation (sleet/snow/freezing rain)
PSR	mixed rain and snow
<i>Tide stage</i>	
TSE	ebb tide
TSF	flood tide
TSH	high tide
TSL	low tide
<i>Wave height</i>	
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 meters
WH4	1.0 to 1.3 meters
WH5	1.3 or greater meters
<i>Wind direction</i>	
N	from the north
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
SW	from the southwest
WSW	from the west southwest
W	from the west
WNW	from the west northwest
NW	from the northwest
NNW	from the north northwest
<i>Wind speed</i>	
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

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

Sensor errors

- SBL Value below minimum limit of method detection
- SCB Value calculated with a value that is below the MDL
- 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

- CCL clear (0-10%)
- 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

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— PDR — drizzle
 — PLR light rain
 — PHR heavy rain
 — PSQ squally
 — PFQ 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 meters
 — WH4 1.0 to 1.3 meters
 — WH5 1.3 or greater meters
 — *Wind direction*
 — N from the north
 — 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
 — SW from the southwest
 — WSW from the west southwest
 — W from the west
 — WNW from the west northwest
 — NW from the northwest
 — NNW from 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 –

Use this section for further documentation of the research data set. Include any additional notes regarding the data set in general, circumstances not covered by the flags and comment codes, or

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~~specific data that were coded with the CSM “See Metadata” comment code. 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 at the sample sites. Include the following excerpt:~~

Data may be missing due to problems with sample collection or processing. Laboratories in the NERRS 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.

Notes:

Data denoted as missing were generally lost as a result of our inability to access a station as a result of acute weather events or safety concerns that occurred during a sampling effort (i.e. not an event that caused the rescheduling of an entire trip) or loss of sample resulting from equipment failure.

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