# VIRGINIA CHESAPEAKE BAY TRIBUTARY WATER QUALITY MONITORING PROGRAM STANDARD OPERATING PROCEDURES MANUAL

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Chesapeake Bay Program
Virginia Department of Environmental Quality
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Richmond, Virginia

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#### **List of Acronyms**

CAR Corrective Action Request
CBM Chesapeake Bay Monitoring
CBO Chesapeake Bay Office
CBP Chesapeake Bay Program
CBPWQ Chesapeake Bay Water Quality

CEDS Comprehensive Environmental Data System
CIMS Chesapeake Bay Information Management System

**CSSP** Coordinated Split Sample Program

**DCLS** Division of Consolidated Laboratory Services

**DEQ** Department of Environmental Quality**DUET** Data Upload and Evaluation Tool

DI Deionized Water
DO Dissolved Oxygen
EDT Electronic Data Transfer

**ETMP** Enhanced Tributary Monitory Program

NRO Northern Regional Office
ODU Old Dominion University
OIS Office of Information Systems
PCN Particulate Carbon and Nitrogen

PP Particulate Phosphorus
PRO Piedmont Regional Office

**PMTF** Procedure Modification Tracking Form

QA Quality Assurance QC Quality Control

SOP Standard Operating Procedure TRO Tidewater Regional Office

**VDEQ** Virginia Department of Environmental Quality

**WQAP** Water Quality Assessments & Planning

**USGS** U.S. Geological Survey

**VDGIF** Virginia Department of Game and Inland Fisheries

**WQM** Water Quality Monitoring portion of the CEDS2000 database program

#### 1.0 PROGRAM PLANNING AND REQUIREMENTS

#### 1.1 SCHEDULING/RESCHEDULING OF CRUISES

Sampling cruises are scheduled into the Water Quality Monitoring Module of the CEDS2000 database (WQM) by the 25<sup>th</sup> of the month prior to the sampling event (refer to the OIS WQM operating manual for specific instructions). Once entered into WQM, the schedule may be modified as needed to accommodate changes due to weather disturbances or equipment malfunctions. Cruises should not be rescheduled less than 2 weeks from the next cruise on that same tributary.

#### 1.1.1 Tributary Cruise Schedule

The tributary cruise schedule is updated annually and distributed to interested parties by DEQ's Tidewater Regional Office. The cruises are typically scheduled as follows:

James River: First Tuesday of the month

Rappahannock River: Second Tuesday of the month

Elizabeth River: Second or third Tuesday and Wednesday of the month.

York River: Third week of the month on Tuesday or Thursday

The scheduling is adjusted around holidays such that cruises are typically not performed the day before or after a holiday. A copy of the current calendar year cruise schedule may be requested from Cindy Johnson (cindy.johnson@deq.virginia.gov).

#### 1.1.2 Rescheduling

A sampling run that does not take place as scheduled <u>must</u> be rescheduled as soon as possible. Any sampling run that is rescheduled 2 days or closer to the scheduled date needs to have the lab notified both by WQM and via e-mail.

- 1. DCLS needs to be notified both through WQM and via e-mail due to time constraints for chemical standards used with their equipment. Send the email to:

  Johnesta.Foneville@dgs.virginia.gov,Shane.Wyatt@dgs.virginia.gov,
  haley.watson@dqs.virginia.gov, jay.armstrong@dgs.virginia.gov,
  Marilyn.bibbs@dgs.virginia.gov, Romin.mustak@dgs.virginia.gov,
  ryan.lewis@dgs.virginia.gov, tariq.mohammed@dgs.virginia.gov,
  terri.harper@dgs.virginia.gov and
  elaine.mason@dgs.virginia.gov. The text of the e-mail should include the region, the
  type of run (i.e. Chesapeake Bay), rescheduled date and the total number of bacterial
  samples that are being rescheduled.
- 2. Phytoplankton samples are collected monthly. Plankton sample pickup needs to be coordinated with ODU during those months. TRO will need to coordinate the plankton sample drop-off with ODU or sample pickup with Central Office in the event they are unable to deliver the samples to ODU.
- 3. Whenever possible, USGS will coordinate sampling of Fall Line stations for baseflow

- with DEQ sampling schedules. CO will contact USGS in the event of any changes to the DEQ schedule.
- 4. The following is an outline of protocols, in order of preference, to be used in the event of a disruption in the sampling schedule:
  - I. Problems due to weather.
    - Option 1 All regions reschedule the run, preferably to the first day when all can sample (see Section 1.4 below for further details).
    - Option 2 Unaffected regions complete sampling as scheduled, affected regions reschedule to first available day.
  - II. Problems due to boat/engine malfunctions.
    - A. If occurring prior to sampling:
    - Option 1 Affected region uses backup boat.
    - Option 2 Unaffected regions adjust their schedules to sample the stations missed by the affected region.
    - Option 3 All regions reschedule the run to the first day when all can sample simultaneously.
    - Option 4 Unaffected regions complete sampling as scheduled, affected region reschedule to first available day.
    - B. If occurring during sampling:
    - Option 1 Unaffected regions sample missed stations on day of sampling.
    - Option 2 Missed stations are sampled on first available day.

If disruptions in sampling occur for any reason, the regional office should notify Cindy Johnson and all labs as soon as possible. Upon completion of scheduled runs, regional offices will report work completed via the Field Summary Sheets, Li-cor data sheets and WQM data sheets (Appendix A). Any deviations from the SOP will be reported via the comment field in WQM and field summary sheets (if only occurs during a single sampling event) or Procedure Modification Tracking Form (if occurs during multiple sampling events or permanent).

5. Notify CBP.

#### 1.2 PERTINENT TELEPHONE NUMBERS

# TRO (757)

647-4140 Cory Routh 647-7642 Nick Black

# **PRO (804)**

659-2695 Matt Carter 720-2229 Roland Whitehead 659-2697 Mike Shaver 659-2708 Warren Smigo 929-7873 Emily Taff

# **NRO**

571-408-1616 Jeff Talbott (o)

# **ODU (757)**

683-4994 Phytoplankton laboratory

# **CBP (804)**

659-2653 Cindy Johnson (o) 334-7590 Cindy Johnson (c)

698-4253 Drew Garey (o) 502-7927 Drew Garey (c)

# DCLS (804)648-4480

ext.328 Jay Armstrong (o)
–CBNUT-3 (NTNP-3 AT
PLANKTON SITES), PNC
and PP:
ext.533 Ryan Lewis(o)
SOLIDS & Chlorophyll
Ext. 266 Johnesta Fonville
(o) Microbiology
Ext. 138 Elaine Mason (o)
Sample Support Services

# 1.3 SAMPLE EXCHANGE, BOAT LAUNCH AND RETRIEVAL SITES

Prior to 1998 sample drop-off sites were used to deliver samples to the participating labs. The drop-off sites are listed in Appendix E and may be used in cases where a DCLS courier is unavailable.

#### Boat launch (L) and retrieval (R) sites are as follows:

#### A. York River

#### PRO:

L/R - Lestor Manor VDGIF ramp at end of Rt. 672 for station 8-PMK034.17

L/R - VDGIF ramp on Mattaponi in town of West Point for station 8-MPN004.39

L/R - Private ramp off route 629, in Walkerton for 8-MPN029.08

#### TRO:

L - Public ramp under Coleman Bridge in Gloucester

R - Game Commission ramp on the Mattaponi River in West Point

#### B. Rappahannock River

#### NRO:

L/R – Private Ramp in River Creek neighborhood.

#### PRO:

- L VDGIF ramp at Gwynn Island
- R Hoskins Creek public boat Ramp

#### C. James River

#### PRO:

- L Chickahominy Riverfront Park, James City County
- R Osbourne Department of Game and Fishery ramp in Henrico Co.

#### TRO:

- L Public boat ramp in Willoughby Bay
- R James City County Marina, Powhatan Creek

#### D. Elizabeth River

TRO: Elizabeth River Park at the Jordan Bridge

#### 1.4 STATION SAMPLING ORDER

Whenever possible sampling is to begin at the mouth of the tributary and proceed sequentially upstream.

The order of priorities for sampling regime should be as follows:

1) Whole river same day,

Whole river sampled upstream sequentially.

2) Whole river same day,

Each region sampling upstream sequentially.

3) Whole river same day,

Most regions (or stations) sampling upstream sequentially.

4) Whole river over several days,

Each day sampling upstream sequentially.

In general, it is understood that the regions sample in the following manner (recognizing the aforementioned sampling priorities):

On the James River # 1 or #4 is observed. Both TRO and PRO are able to sample in time sequencing fashion, sampling from downstream to upstream order.

On the York River # 3 is observed. Since the PMK and MPN station sampling involves 3 trailer launches, PRO also has always sampled them in downstream order to finish the run before dark.

On the Rappahannock River # 3 is observed. Safety considerations prevent NRO from sampling their segment of the Rappahannock River sequentially to PRO but NRO does sample their segment of the Rappahannock sequentially upstream.

#### 1.5 STATION LOCATIONS (NAD83)

Station latitudes and longitudes listed are those of current sampling stations. Some may differ from the legacy Storet database latitudes and longitudes.

Α.	<b>Stations</b>	samp	led 1	by 1	PRO
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River Basin Station Name	Latitude	Longitude	Latitude	Longitude
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	River Mile	CBP Format	(deg.,min.,sec.) NAD83	(deg.,min.,sec.) NAD83	(decimal deg.)	(decimal deg.)
	3-RPP010.60	LE3.4	37° 37' 54.8"	-76° 26' 41.5"	37.63189	-76.44486
	3-CRR003.38 (Corrotoman River)	LE3.3	37° 41' 18.3"	-76° 28' 27.9"	37.68842	-76.47442
Rappahannock River	3-RPP017.72	LE3.2	37° 40' 08.9"	-76° 33' 01.7"	37.66914	-76.55047
(Run ID:	3-RPP025.52	LE3.1	37° 45' 33.3"	-76° 36' 57.3"	37.75925	-76.61592
PRAP1)	3-RPP031.57	RET3.2	37° 48' 41.7"	-76° 42' 43.0"	37.81158	-76.71195
	3-RPP042.12*	RET3.1	37° 55' 02.3"	-76° 49' 19.9"	37.91730	-76.82220
	3-RPP051.01*	TF3.3	38°01' 06.5"	-76° 54' 33.4"	38.01847	-76.90928
	2-CHK006.14 (Chickahominy River)	RET5.1A	37° 18' 44.3"	-76° 52' 36.2"	37.31230	-76.87672
	2-JMS055.94	TF5.6	37° 16' 21.8"	-76° 59' 26.1"	37.27272	-76.99057
	2-JMS069.08	TF5.5A	37° 18' 05.9"	-77° 07' 42.2"	37.30165	-77.12840
James River	2-JMS075.04*	TF5.5	37° 18' 45.5"	-77° 13' 58.1"	37.31265	-77.23283
(Run ID: PJAM1)	2-APP001.53 (Appomattox River)	TF5.4	37° 18' 44.64"	-77° 17' 28.78"	37.31240	-77.29133
	2-JMS099.30	TF5.3	37° 24' 11.2"	-77° 23' 33.8"	37.40310	-77.39272
	2-JMS104.16	TF5.2A	37° 27' 00.0"	-77° 25' 07.8"	37.45000	-77.41883
	2-JMS110.30	TF5.2	37° 31' 49.8"	-77° 26' 02.4"	37.53050	-77.43400
York River	8-MPN004.39 (Mattaponi River)	RET4.2	37° 34' 16.5"	-76° 47' 49.7"	37.57125	-76.79715
(Run ID:	8-MPN029.08	TF4.4	37°43'22.1"	-77°01'32.38"	37.7228	-77.02566
PYRK1)	8-PMK034.17* (Pamunkey River)	TF4.2	37° 34' 48.0"	-77° 01' 16.6"	37.57999	-77.02128

<sup>\*</sup> Denotes plankton site

B. Stations sampled by NRO

	<u> </u>					
	Station Name		Latituda	T 14 1-	Latitude	Longitude
River Basin	River Mile	CBP Format	Latitude (deg.,min.,sec.)	Longitude (deg.,min.,sec.)	(decimal deg.)	(decimal deg.)
	3- RPP064.40	TF3.2A	38°06' 46.6"	-77° 03' 17.4"	38.11295	-77.05482
	3- RPP080.19	TF3.2	38° 10' 28.8"	-77° 11' 11.8"	38.17466	-77.18661
Rappahannock (Run ID: N24 CBT)	3- RPP091.55	TF3.1B	38° 14' 46.4"	-77° 14' 00.4"	38.24622	-77.23344
1N24_CD1)	3- RPP098.81	TF3.1E	38° 14' 40.9"	-77° 19' 30.3"	38.24469	-77.32508
	3- RPP106.01	TF3.1F	38° 16' 10.3"	-77° 25 44.3"	38.269527	-77.428972

C. Stations sampled by TRO

•	Station Name				Latitude	Longitude
River Basin	River Mile	CBP Format	Latitude (deg.,min.,sec.)	Longitude (deg.,min.,sec.)	(decimal deg.)	(decimal deg.)
	2-ELI002.00 (Elizabeth River)	LE5.6	36° 54' 16.4"	-76° 20' 18.1"	36.90456	-76.33836
Elizabeth	2-LAF001.15	LFA01	36°54'29.6"	-76°18'52.7"	36.9082	-76.3146
River I	2-LAF003.83	LFB01	36°53'21.7"	-76°16'53.2"	36.8894	-76.2814
(Run ID:	2-ELI003.52	ElI2	36°52'54.8"	-76°20'19.3"	36.881888	-76.338694
TELRIA)	2-ELI004.79	ELD01	36°51'56.10"	-76°19'44.3"	36.86556	-76.32899
	2-WBE002.11	WBE1	36°50'38.4"	-76°21'45"	36.84400	-76.360805
	2-WBE004.44	WBB05	36°49'45"	-76°23'45"	36.82917	-76.39583
Elizabeth River	2-ELI006.92	ELE01	36°50'53.98"	-76°17'53"	36.84833	-76.29806
II	2-EBE002.98	EBB01	36°50'10"	-76°14'40"	36.83611	-76.24444
	2-EBE000.40	EBE1	36°50'27.6"	-76°17'16.8"	36.84100	-76.28800
(Run ID:	2-SBE001.98	SBE2	36°48'48.2"	-76°17'25.1"	36.813388	-76.29030
TELRIB)	2-SBE006.26*	SBE5	36°45'54.7"	-76°17'59.6"	36.765194	-76.29988
	2-JMS005.72	LE5.4	36° 57' 17.5"	-76° 23' 33.9"	36.95486	-76.39275
James River	2-JMS013.10	LE5.3	36° 59' 25.6"	-76° 28' 31.6"	36.99044	-76.47544
(Run ID:	2-JMS021.04	LE5.2	37° 03' 21.6"	-76° 35' 35.0"	37.05600	-76.59306
TJAM1)	2-JMS032.59	LE5.1	37° 12' 10.7"	-76° 38' 54.0"	37.20297	-76.64833
	2-JMS042.92*	RET5.2	37° 12' 10.6"	-76° 46' 55.9"	37.20294	-76.78219
York River	8-YRK001.64	LE4.3	37° 14' 02.1"	-76° 25' 51.4"	37.23392	-76.43094
(Run ID: TYRK1)	8-YRK011.14	LE4.2	37° 17' 25.6"	-76° 34' 41.2"	37.29044	-76.57811
	8-YRK022.70	LE4.1	37° 25' 07.8"	-76° 41' 28.5"	37.41883	-76.69125

8-YRK031.39*	RET4.3	37° 30' 31.3"	-76° 47' 20.0"	37.50869	-76.78889
8-PMK006.36 (Pamunkey River)	RET4.1	37° 31' 32.3"	-76° 52' 03.4"	37.52564	-76.86761

<sup>\*</sup> Denotes plankton site

#### 1.6 PLANKTON SAMPLE PICKUP/DROP-OFF

#### **Sample Delivery**

PRO and ODU will arrange sample pickup as needed.. TRO will deliver the samples to room 244, in the Department of Ocean, Earth and Atmospheric Sciences building. All delivery times need to be confirmed with ODU Phytoplankton Lab personnel on that day. The Lab phone is 757-683-4994. In emergencies, and in instances when it is not possible to deliver the cooler on time, contact the Phytoplankton Lab. If there is a HAB emergency follow the procedures outlined in the DEQ HAB decision tree.

Directions for PRO sample pickup/delivery are as follows:

# Rappahannock River

ODU meets PRO at the Hoskins Creek public boat ramp in Tappahannock at 1:00 pm and returns with samples to ODU (approximate drop-off time 5:00-5:30 pm)

The Hoskins Creek boat ramp is located at the end of Dock St. off of Route 17 in Tappahannock Total Driving time: approximately 3 hours

#### York River:

ODU meets PRO at the Lestor Manor public boat ramp at end of Rt. 672 at 11:30 am Directions (King William County Map):

The public boat ramp at the end of Rt. 672 is the final road on the left on Rt. 633 before entering the Pamunkey Indian Reservation

Total Driving time: 2 hours 15 minutes

#### **James River:**

ODU meets PRO at Hopewell City Ramp off Route 10 at 12:30pm

The Marina is straight across from the end of Riverside Loop Dr.

Driving time: 2 hours 15 minutes

#### 2.0 PRE-CRUISE PREPARATIONS

In preparation for a sampling run where field measurements are to be taken, be sure that operating manual instructions have been followed concerning preventive maintenance and calibration for all equipment to be used. Where possible, backup instruments and/or sample collecting strategies should also be prepared and taken in the field (see Section 2.7).

#### 2.1 PNC FILTER MUFFLING

25 mm diameter, 1.0 µm pore-sized, Cytiva Whatman glass fiber filters (GFF) are used for sample filtration. To prevent re-contamination of the muffled filters do not carry them over from month to month. Muffle fresh filters for the first cruise of each month and throw out any unused filters after the last cruise of the month.

- 1) Acid wash aluminum foil tray or porcelain crucible with 10%N HCL or heat to 1,000 °C to sterilize. Allow crucible to cool in desiccator dish.
- 2) Allow muffle furnace to cool to 500 °C or preheat to 500 °C.
- 3) Pre-determine the number of filters needed for the particular field event and place them on aluminum foil tray or in crucible. One filter is required for each sample to be obtained and two additional dry muffled filters are required by DCLS for the determination of background carbon content. Spread the filters around the tray or crucible as much as possible to allow filters to cook evenly.
- 4) Place the aluminum foil tray or crucible containing the filters in the muffle furnace, close and latch the door.
- 5) Heat at 500 °C for 60 minutes.
- 6) After 60 minutes, remove the filters from the furnace and allow them to cool in a desiccator for several hours.
- 7) Place the filters in a plastic snap lid box with desiccant.

#### Replace desiccant as needed (usually when the blue color of the gel lightens to pink or white).

Important points regarding filters:

- Muffling time and oven temperature are critical do not over-bake or under-bake the filters.
- ♦ It is advisable to muffle extra filters to allow the filtration of additional sample in the case of accidental loss.

# 2.2 MULTI-PARAMETER INSTRUMENT CALIBRATION

Multiprobes should be calibrated on the morning of each run and checked for drift upon completion of each run following the procedures outlined in the DEQ WQM SOP and those of the manufacturer. Up-to-date instruction manuals for each multiprobe should be kept on file at each regional office.

#### **QA/QC** Criteria:

# a. Specific Conductance

All specific conductance readings must be within  $\pm$  5% of the standard.

#### b. pH

Readings must be within  $\pm 0.2$  SU of each standard.

#### c. Dissolved Oxygen

Theoretical and actual DO saturation values should be within  $\pm$  0.29 mg/L.

Notes on DO calibration and QA/QC procedures:

Follow the manufacturer's recommended procedure for calibrating DO based on simulated saturation conditions (water-saturated air or air-saturated water method). Note the barometric pressure and temperature. Compare the theoretical DO at the observed temperature and pressure (Appendix B) to the sonde value and calibrate such that the actual value equals the theoretical value.

#### d. Depth

The depth needs to be calibrated in the field just prior to sampling the first station. See Chapter 3 for details.

#### e. Temperature

Central office personnel will conduct temperature checks for multiprobes against an NIST certified thermistor annually when conducting site visits.

Regions should check the temperature probe against another multiprobe instrument's temperature probe semi-annually. If a discrepancy should occur (temperatures are not +/-1 °C) contact Central Office so that the probes can be checked against an NIST certified thermistor as soon as possible. If there is good agreement between the instruments, then Central Office personnel will check the instruments against an NIST certified thermistor as planned.

- 1.The temperature check should be conducted in an ice/water mixture (approximately 4 °C) and at a warm water temperature that will best approximate the highest ambient temperature expected to be sampled (approximately 30 °C). The probe(s) and/or NIST certified thermistor are lowered into the mixtures simultaneously, allowed to stabilize, and read.
- 2. Send the multiprobe unit back to the manufacturer for temperature calibration if the thermistor and multiprobe readings differ more than  $0.5~^{\circ}$ C.

#### f. Barometric Pressure

Compare the instrument Barometric Pressure (BP) value to the barometric pressure in mm Hg read from a NIST traceable barometer. If a NIST traceable barometer is not available, the nearest National Weather Service or NOAA weather station barometric pressure readings may be used.

If the difference between the reading and the traceable barometer is greater than 10 mmHg, the Surveyor needs to be calibrated to the traceable barometer.

For calibration, a corrected barometric pressure should be obtained from a barometer (mm Hg) or

from the local weather bureau (inches Hg). Inches of Hg are converted to mm Hg by multiplying by 25.4. Plug the corrected barometric pressure from the barometer or local weather bureau in mm Hg into the following formula to obtain the uncorrected BP that will be used in the calibration: uncorrected BP = corrected BP-2.5( $A_{\rm ft}/100$ ), where  $A_{\rm ft}$ = altitude in feet above mean sea level.

#### PREPARATION FOR USE

- 1) If the short calibration cable is used for calibration, switch the calibration cable to a longer cable.
- 2) If a modified calibration cup is used for calibration to conserve standards, remove it from the sonde and screw on the storage cup filled with sufficient pH 4 buffer or tap water (~10 ml). Do not immerse the pH sensor. Change over to the sensor guard for transport/use in the field.

# 2.3 CLEANING FILTRATION EQUIPMENT:

Take down all towers, graduated cylinders and forceps and put in the lab for cleaning.

Observe all safety precautions.

All safety measures, necessary with the use of acid <u>MUST</u> be enforced. This includes the use of skin protection, such as rubber gloves, full-face shield, apron and footwear, as well as cleaning the equipment under a fume hood.

Clean one piece at a time.

Wash, rinse and strip all towers and graduated cylinders.

Wash all towers with a phosphate free detergent such as Liquinox®. Rinse all towers with tap water, strip them with 25 mL of 10% HCl and rinse 3 times again with DI water. Wash, rinse and strip the graduated cylinders in the same manner as the towers using approximately 10% of the total volume of the graduated cylinder of 10% HCl. A brush may be needed to remove any remaining sediment.

Rinse all utensils with DI water 3 times (e.g. forceps). Do not use acid on metal utensils.

Water will be trapped in the filtering apparatus. Use the flexible tube from the water collection tank to suck up water from inside the towers.

Store the clean filtration equipment in a manner that prevents contamination (e.g. covered in aluminum foil or plastic).

#### 2.4 CLEANING THE LI-COR SENSORS:

The Li-cor sensors must be kept clean from scum and hard water deposits. Prior to each cruise clean the Li-cor sensors with a soft cloth or sponge, water and mild diluted detergent (such as Liquinox®). **Be sure not to scratch the surface of the sensor.** A small amount of undiluted vinegar can be used to remove hard water deposits from the diffuser element.

#### 2.5 FILTERS AND DI WATER:

- 1. 47 mm diameter, 0.7 μm pore size glass Whatman GF/F filters (per station, one filter will be needed per PP sample and 1-3 per chlorophyll sample depending on water turbidity).
- 2. 25 mm diameter, 1.0 µm pore size Gelman type A/E or Whatman GFF (pre-muffled) for PNC (one per sample). **Reminder**: PNC filters are to be prepared fresh at the beginning of each month.
- 3. Fill two 5-gallon Carboy bottles with fresh DI water for equipment blanks and carry 1-2 gallons of DI to rinse the filteration equipment between sites. One to 1.5 carboy(s) is needed to clear the hose and the remainder for filling containers,
- 4. A spare container of DI water should also be kept in the vehicle in case needed.

#### 2.6 SAMPLE BOTTLE PREPARATION:

#### A. All Stations

All 250 mL and 2 L brown bottles and milk jugs (new and reused) need to be detergent washed, acid rinsed with 10% HCL and triple rinsed with DI water prior to their use in the field. New  $\frac{1}{2}$  gallon cubitainers do not need to be pre-washed in the lab.

- 1. Bottles needed per station:
  - 2 acid washed and rinsed 250 mL CBNUT-3 (NTNP-3 AT PLANKTON SITES) bottles
  - $1 \frac{1}{2}$  gallon cubitainer sample container for surface solids analysis (NME7)
  - $1 \frac{1}{2}$  gallon cubitainer sample container for bottom solids analysis (NME7)
  - 2 acid washed and rinsed 2L HDPE brown bottles or pre-cleaned (see above) milk jugs (for bottom only as Chl-a samples need to be kept in the dark)
- 2. 1 2 complete extra sets of bottles in case of contamination or mishap.
- 3. Bottles needed for QA/QC procedures:
  - a. Equipment blanks:
    - 1 acid washed and rinsed 250 mL CBNUT-3 (NTNP-3 AT PLANKTON SITES) bottle
    - 1 ½ gallon cubitainer for solid analyses
    - 1- acid washed and rinsed 2 L brown HDPE bottle for filters and filtrate analyses
  - b. Duplicate samples:
    - 1 acid washed and rinsed 250 mL CBNUT-3 (NTNP-3 AT PLANKTON SITES) bottle
    - $1 \frac{1}{2}$  gallon cubitainer for solid analyses
    - 1 acid washed and rinsed 2 L brown HDPE bottle for all filter and filtrate analyses
- 4. Bottles needed per-station at phytoplankton sites
  - 2 500 mL bottles with Lugol's preservative
  - 2 250 mL HDPE bottles

#### 2.7 PETRI DISHES, SHEETS AND TAGS:

#### Per station:

- 1 WOM Field sheet
- 9 sample tags
- 4 Petri dishes
- 1 square Aluminum foil

#### QA/QC:

#### Filtration blanks

- 2 Petri dishes
- 4 Sample tags
- 1 square aluminum foil

# Equipment blanks

- 2 Petri dishes
- 5 sample tags
- 1 square Aluminum foil

#### Duplicate samples

- 4 Petri dishes
- 5 sample tags
- 1 square Aluminum foil
- PNC and PP samples are to be collected in petri dishes. Do Not Use foil for PNC samples. Each

- sample is to be labeled with a sample tag with station, date, depth, unit code, collector's initials, group code and container #. When using labels to seal the petri dishes be sure the label is situated such that it can be clearly read by laboratory personnel.
- Aluminum foil is used for chlorophyll-a samples, clearly mark each sample and place labels on the foil so the group code is clearly visible to sample support personnel at DCLS. Do not filter more than 3 filters per station for chlorophyll-a.

Take 10 extra Petri dishes or aluminum foil squares, sheets and tags in case of contamination or mishap.

#### 2.8 COOLERS AND TEMPERATURE TESTING BOTTLES

- Take enough ice in coolers to cool samples to 4° Celsius (+/- 2° C) and maintain them at that temperature. If necessary, place additional ice on the samples upon returning to the regional office, draining excess water from the coolers. Sample temperatures must be 4° Celsius (+/- 2° C) when processed by DCLS Central receiving personnel.
- A bottle of colored water (usually pink or red) for temperature testing must be placed in each cooler prior to sample collection and must remain in the cooler for delivery to DCLS. Samples in coolers not containing the colored water bottles and those for which water is found not to be at < 6° C will be rejected.

# 2.9 BACKUP SAMPLING EQUIPMENT

Whenever feasible, backup equipment should be taken in the field for use in the event of problems with sampling gear, such as a multiprobe or water pump failure.

The following is a list of suggested equipment that should be available if problems occur:

- Backup multiprobe unit (if available), calibrated according to the DEQ Agency QAPP/SOP
- Backup water pump system (if available)
- An alpha water sampling bottle with spare messenger
- Thermometer
- A Winkler sampling kit when a backup multiprobe unit is unavailable
- Backup filtration unit
- Spare deep cycle batteries to run filtration unit.

Prior to the scheduled day of sampling, each region must also make sure that there are adequate supplies of coolers, ice, cubitainers, chlorophyll bottles, sample data sheets, sample tags, and indelible pens.

# 3.0 FIELD PROCEDURES

#### 3.1 FIELD DOCUMENTATION

All data sheets discussed in this section are included in Appendix A. Regional offices should place electronic copies of Field Summary Sheets, Li-cor data sheets and all WQM Field data sheets in the Region's sharepoint folder as soon as possible after cruises from May – September ( when public interest is highest in dissolved oxygen data). All documentation from October-April cruises should be scanned and e-mailed once per month.

#### 3.1.1 Field Summary Sheet

- 1. One Field Summary Sheet will be filled out per sampling run.
- 2. The following information will be entered on the Field Summary Sheet.
  - Date
  - Stations sampled
  - Time vessel reached station
  - Latitude and Longitude of starting point on station. Use GPS to ensure sampling occurs preferably within 25 m of the field coordinates listed in this SOP but no further than 37 m (0.02 nautical miles) away from the site.
  - Comments, problems or unusual events encountered on station
  - Multiprobe post cruise calibration check information (performed after sampling). If this information is not included on the field summary sheet, the RO must send the post cruise information to Central Office for QA/QC verification.

#### 3.1.2 WQM Field Data Sheet

- 1. Each station will require one WQM data sheet, so multiple data sheets will be required per run.
- 2. All field measurements and water sample information are entered onto this sheet.
- 3. Field personnel must fill in the following items with indelible ink:

#### a. General Information:

#### Be sure this matches sample tag exactly!

- Date (mmddyyyy)
- Time (24-hr military format)
- Station description (Station name in river mile format. See pages 5-7).
- Analytical Group Code (e.g NTNP-3)
- Container Number
- Sample depth (m)
- Volume filtered (for filtered parameters)

#### **b.** Field Parameters:

- Weather code: 1 = cloudy, 2 = precipitation, 3 = clear, 4 = fog
- Tide code: 1 = high, 2 = low, 3 = flood, 4 = ebb
- Secchi depth: in 0.1 m increments. Do not list as > 3.0 or 3.0 +. Attach additional marked line if the visibility is greater than the length of line attached to your Secchi disk.
- -Bottom depth (m): In the upper right side of the Field Data sheet indicate the true depth of the station (e.g. 8.7 m) as obtained from the multiprobe/data sonde.

#### c. Vertical Profile:

Fill in the datasonde probe values for temperature, specific conductance, salinity, pH and DO at each depth sampled. Allow ample time for each parameter to equilibrate at each depth prior to recording a value. Record values to the hundredths place.

#### 3.1.3 DCLS Laboratory Submission Form

- DCLS Laboratory submission forms must be completed for samples not included in WQM (CEDS). This will be rare and will likely only occur when the CEDS database is down/unavailable.
- 2. Be sure the following information matches the sample tags exactly:
  - Station Id (DEQ Rivermile format)
  - Date (mmddyyyy)
  - Time (24-hr military format)
  - Sample depth (m)
  - Analytical Code
  - Container Number
  - Collector's initials

# 3.1.4 LI-COR Light Attenuation Sheet

This data sheet will be used to record all Li-cor measurements and comments pertinent to Li-cor readings obtained on a sampling run.

#### 3.1.5 Field Filtration Log

#### All nutrient parameters must be filtered within 2 hours of their collection.

Regions not immediately filtering on board the boat or recording time filtered on their WQM field sheets should use this form to record individual sample filter times.

#### 3.2 FIELD MEASUREMENTS

Field measurements to be obtained include the following:

#### 3.2.1 Secchi Disk

- 1. Use a 20 cm diameter Secchi disk attached to a line or chain marked in 0.1 m increments with paint or tape. **Check marks annually for accuracy.**
- 2. Lower the Secchi disk into the water on the **shaded** side of the boat until the black and white quadrants are no longer distinguishable. Attach additional marked line if the visibility is greater than the length of line attached to your Secchi disk. **Do not wear sunglasses while obtaining this reading.**
- 3. Note the depth at which the quadrants were no longer distinguishable and then raise the disk until the quadrants are again distinct.
- 4. The recorded Secchi depth is the average of the two depths to the closest 0.1 m. If the Secchi disk is still visible when it reaches the bottom, record the bottom depth as secchi depth and insert a comment in the comment field to indicate secchi disk was lying on the bottom.

#### 3.2.2 Vertical Profile

Measure a vertical profile of temperature, dissolved oxygen, conductivity, salinity and pH using a Calibrated multi-parameter water quality monitoring instrument (such as a YSI brand water quality monitoring system).

#### Notes:

- The multiprobe must be calibrated and the operation checks must be performed prior to using it in the field (See Section 2.2).
- While the unit is in operation aboard the vessel, the boat operator must maintain the boat's position and orientation with wind and tide movement to ensure that the sonde hangs as vertically as possible.
- While the unit is in operation aboard the vessel, **keep the probe away from the engine's propeller** to ensure the safety of the sonde and data cable, and to prevent interference from the propeller wash with all water quality measurements.
- -Field personnel should wait as long as possible prior to collecting water samples after ships or barges pass to ensure they do not sample any bottom sediment that the ship may have suspended into the water column.
- -Regional personnel have agreed to conduct Steps 4, 5, 6 and 13 below whenever the previous DO post cruise calibration checks for the instrument in use is out of range.
  - 1. At least 5 minutes prior to arriving at the first station <u>remove unit from storage</u>.
  - 2. Replace the storage cup with the sensor guard (datasonde).

    Remove the storage cup from the sonde and screw on the sensor guard in its place.

    Caution should be taken to avoid any contact with the sensors.
  - 3. Connect the two ends of the data cable to the probe.

    Ensure that the probe is attached to the data cable with the pin and clip ring.
  - 4. Wrap the sensor guard in a moist towel so that area containing the sensors is completely covered and place in a bucket.
  - 5. Turn the instrument on and allow it to stabilize.
  - 6. If the previous post cruise calibration check for DO was out of range, confirm the DO calibration:

#### Check the DO percent saturation

Prior to collecting the vertical profile while on station check the DO percent saturation. The probe unit should remain wrapped in the moist towel for this calibration check. Percent saturation should read between 95-105%. If not, the instrument should be calibrated for DO (while in the moist towel) before taking the profile.

- 7. Calibrate depth.
  - a. If using a Datasonde or Minisonde: Point the sonde upward.
  - b. Turn the instrument on
  - c. Press "setup/cal", "calibrate", "sonde" and "depth"
  - d. Use the left, right, up and down arrows to calibrate to 0.0 depth.
  - e. Press "enter" and "Y" to save.

# 8. Lower the sonde to 1 meter above the bottom sediment.

Lower the sonde with cable and weighted nylon safety line (if used) through the water column. Continue lowering the sonde until the weight touches the bottom (if a weight is

used the sonde sensors are 1 m above the bottom). Alternately, lower the sonde until it touches the bottom. Record the true total depth in the upper right corner of the field sheet. Round the true depth to the nearest whole number and raise the sonde to the next whole meter above that. For example, if the true depth is 6.6m, then the depth would be rounded to 7m and the bottom sample would be collected at a depth of 6m. If the true depth were 5.2m, then the depth is rounded to 5m and the bottom sample would be collected at 4m. If the true bottom depth happens to fall on a 0.5m increment then round down (i.e. 5.5m rounds to 5m and the bottom sample is collected at a depth of 4m).

- 9. Wait for thermal equilibrium.
  - Allow approximately one minute for thermal equilibrium, and then verify that all readings are stable.
- 10. Record each parameter reading on data sheets.
  - View the display unit for each parameter (temperature, pH, DO, conductivity, salinity and depth). Record each parameter on the WQM data sheet. Be sure each parameter has stabilized prior to recording a value on the field sheet. Measurements for temperature, DO and pH should be recorded to the hundredths place and specific conductance recorded in whole numbers.
- 11. Record measurements at each meter to 1 meter below the surface.
- 12. After all measurements have been collected at each required depth, retrieve the sonde while coiling the cable to prevent tangles. When the sonde has reached the surface, grasp the sonde with one hand while holding the cable with the other hand. Store the sonde in a cool place, keeping the sensors moist. Proper short-term storage methods include placing the sensors and guard in a bucket of ambient water, wrapping them in a damp towel, or replacing the calibration cup, filled tap water. NOTE: sensors should NOT be stored in DI water.

## 3.2.3 Light Attenuation

Optimally, rotation of Li-cor sensors should occur such that each is factory-calibrated yearly. At a maximum each should be calibrated every two years. The Photo Diodes will degrade even when the sensors are not deployed in the field therefore each region will need to track purchases and recalibration dates, along with the dates sensors are used in the field using a LI-COR sensor tracking sheet (Appendix A). The tracking sheets should be kept in a logbook at each region.

The Li-cor instrument requires an air or water multiplier for each sensor depending on the media in which it will be used. The multiplier is the calibration coefficient for each sensor and is specified on the calibration documentation for each sensor. While the multipliers are stored in the data logger and do not need to be entered prior to each use, a new multiplier is required when the sensors are replaced or recalibrated.

#### A. Entering multipliers/ initial set up for LI-1400/LI-1500:

- 1. With the front panel face up and the display at the top, connect the light sensor into the BNC connector I1 located on the top left of the LI-1400 unit and the underwater sensor into the BNC connector I3 located on the top right of the LI-1400 unit.
- 2. Turn on the instrument by pressing the key labeled ON.
- **3.** Press the key labeled **SETUP** on the data logger.

- **4. Set Up Channels** should be displayed. If set up channels is not displayed, use the left or right arrow key to toggle the choices until it is displayed. Press **ENTER**.
- **5.** Using the right or left arrow keys select **I1=light**. Press **ENTER**.
- **6.** Pressing the shift key to access the alpha characters on the numeric key pad (press shift once for the upper character or twice for the lower character) type AIR for the description. Press  $\downarrow$ .
- 7. Type in the multiplier for use in air from the tag attached to the air sensor or from the most recent certificate of calibration. Press \( \preceq \).
- **8.** Using the shift key to toggle between the upper and lower characters on the key pad, type **SA** for the label. Label is a two character alpha numeric code.
  - **Channel 1 is SA** for surface air, **Channel 2 is UU** for underwater up. Press ↓.
- **10.** Use the right or left arrow to set the Log Routine to **none**.
- 11. Press **Esc** twice to return to the setup menu.
- **12. Set Up Channels** should be displayed. Press **ENTER**.
- **13.** Use the down arrow to select **I3=light**.
- **14.** Repeat steps 6-10 to enter the description, multiplier and label (make sure you enter the correct multiplier for its intended use).
- **15.** Press the **View key** and use the left and right arrow to toggle to New Data. Press **ENTER**.
- **16.** Use the left and right arrow key to toggle the display until channel III is displayed. The LI-1400 is now configured to display the running average of the 5 previous seconds' instantaneous values of the quantum sensor and is now ready for use.

#### **B. Data Collection:**

- 1. Li-Cor measurements need to be collected **year round** at plankton/productivity sites.
- 2. Visually inspect Li-cor meter probes and connections.
- Check battery level and ensure probes are positioned properly on deck and subsurface mountings.
- 3. <u>Connect the deck sensor cable to the BNC connection on the top left of the data logger.</u>
- 4. <u>Secure the small deck sensor in an unobstructed and completely unshaded area on</u> the vessel.
- 5. Run the end of the cable for the underwater sensor to the data logger and connect the end of the underwater sensor to the BNC connector on the top right of the data logger.
- 6. <u>Attach sufficient weight</u> to the underwater sensor frame such that the sensor remains upright as it is lowered to depth.
- 7. On the sunny side of the boat, <u>lower underwater sensor to depth just below the surface ensuring that the probe will not rise out of the water with wave action (Note: the depth for this reading is recorded as 0.1 meter in WQM).</u>

- 8. Turn the instrument ON.
- 9. Obtain profile. Report values in whole numbers except for the last depth of the profile, for this observation report values in tenths.
  - At each depth a light attenuation reading is obtained from the deck sensor as well as from the water sensor.
  - Take initial readings with the deck sensor and just below the surface with water sensor.
  - Take second water sensor reading at depth of 0.5 meters.
  - Take successive water sensor readings at 0.5 meter increments.
  - Continue the profile until the underwater sensor displays either a value of approximately 10 micro Einsteins or 20% of the surface light reading.
  - Allow a minimum of 5 seconds between readings (to create a 5-second average value).
  - If the Li-cor instrument is being used as a data logger, depress the "enter" key with each reading.
- 10. When profile is complete, turn the instrument OFF.
- 11. <u>Record data on Li-cor Attenuation Sheet</u> and transmit to CBO at the end of the month or along with the other field documentation sheets.

# 3.3 WATER QUALITY SAMPLE COLLECTION PROCEDURES

#### 3.3.1 Sample Collections

#### A. Water Quality Samples

Each region should have a pump and hose assembly permanently attached to their primary sampling boat. The intake hose must be long enough to collect bottom water samples from the deepest stations, and have enough weight attached to ensure vertical deployment even in strong currents. Samples will be collected 1 m above the bottom (where bottom depth is rounded to the nearest whole-number) and 1 m below the surface.

#### **Hose Clearing Times:**

It is imperative that sufficient hose clearing time be allowed to ensure the water being sampled is obtained from the intended depth. This can be accomplished using the air plug method described below:

#### Air Plug method:

- a. Turn the pump on with the draw end of the hose above the water surface to place air into the hose.
- b. Turn off the pump and lower the hose to the bottom sample depth.
- c. Turn on the pump and watch for the air to completely exit the hose then begin sampling.
- d. Pull hose completely out of the water column and turn pump back on to pull air plug

into the hose.

- e. Turn pump off and lower hose to 1 meter depth.
- f. Watch for the air to completely exit the hose, wait an additional 30 seconds then begin sampling.

#### **Sample Collection:**

- 1. Bottom samples will be obtained first.
- 2. Clear the hose using the air plug method at each sample depth prior to collecting any samples. The hose must be pulled completely out of the water between depths.
- 4. Rinse each sample container twice with sample water before filling. This is especially important when containers are blown open by mouth.
  - Cubitainers are kept closed until immediately before filling. Do not use any container that appears dirty, or appears to have been previously used.
- 5. Fill the following containers to nearly full and cap (note: 250ml bottles are not used here, but will be filled during filtration and submitted for CBNUT-3 analysis).
  - Four to five samples are taken at each station, they are:

Bottom: One Nutrient Sample (one 2-liter HPDE brown bottle previously acid washed and rinsed with DI water)

One whole water Solid Sample for TSS, VSS and FSS (1/2 gallon)

Surface: One Nutrient/Chlorophyll Sample (one 2-liter HPDE brown bottle previously acid washed and rinsed with DI water).

One whole water sample for the Solid Sample (1/2 gallon cubitainer for TSS, VSS and FSS)

- To avoid contamination, minimize contact with the container mouth, inside surfaces, and inside of cap.
- Grasp the cap and pull the neck portion of the cubitainer out to facilitate filling. An ample flow of water from the pump hose should help open the cubitainer.
- Leave some air in the containers to allow better mixing in the bottles
- 6. Attach the appropriate sample tag to the cubitainer before it is put in a cooler surrounding the container with ice to the bottom of the cap.

Place a sample tag on the sample with the following information:

- a) Station
- b) Date (mmddyyyy) and time (24-hour military schedule) of collection (where all samples collected from a station will have the same collection times)
- c) Depth of collection (m)
- d) Unit code
- e) Collector's initials
- f) Group code
- 7. Make sure the cap is tight enough to prevent ice water from contaminating the sample.

# **B. Plankton and Dissolved Organic Carbon Samples**

SOP for the collection of plankton and productivity samples provided by Old Dominion University Phytoplankton Analysis Laboratory

#### Collection gear and bottles:

A. 2 large carboys, Pump with attached hose, Secchi disk, Cooler with ice.

- B. For each station:
  - 2 500 mL bottles with Lugol's preservative
  - 2 125 mL bottles with glutaraldehyde
  - 2 40 mL glass vials for DOCFF (collected at phytoplankton stations only)

#### Plankton Samples:

#### On Station: (Photic zone)

- 1. Take Secchi disk reading, and multiple by 3.5 to obtain photic zone depth.
- 2. Water samples will be taken between this depth and the surface.
- 3. Divide this depth by 5 or use this number to determine from the chart (Appendix B) how far the hose will be pulled up after pumping water into each carboy.
- 4. Lower hose to the photic zone depth, allow water to be pumped through the hose for at least 1 minute. Then pump approximately 3 liters into each carboy.
- 5. Remove hose from carboy, raise hose one increment, and repeat the procedure.
- 6. When completed, agitate the carboys to mix the contents, fill 1 500 mL bottle (containing Lugols) and 1 125 mL (containing glutaraldehyde), from one carboy, and 1-500 mL bottle and 1-125 mL bottle from the second carboy.
- 7. Be sure to record station number and date on all bottles.
- 8. The phytoplankton samples may be stored on ice, however it is not required due to the chemical preservation of the sample.

Discard any water left in the carboys.

#### Collection of Dissolved Organic Carbon (DOCFF) Samples:

#### **Supplies needed:**

- Pre-cleaned and assembled vacuum pump and battery (see CBP SOP Section 2.3)
- Forceps
- One 47 mm diameter 0.7 µm pore sized Whatman GF/F filter per station
- One 47 mm diameter 0.7 μm pore sized Whatman GF/F filter each for QC field replicate and equipment blank (as needed)
- One 250 mL bottle per site
- One 250 mL bottle for filter blank (as needed)
- One 250 mL bottle for Replicate (as needed)
- One 250 mL bottle for NTNP/CBNUT-3
- One or two additional 250 mL bottles for QA/QC samples (as needed)
- Sulfuric Acid as a preservative.
- The use of Non-powdered nitrile gloves is recommended by the Bay Program when filtering for low level nutrients although this is not required by the agency QAPP.

#### **Procedure:**

- 1) Samples are to be collected from surface depths (i.e. 1 meter) at sites monitored for plankton.
- Pre-clean all sample bottles and filtration equipment following the appropriate CBP protocols prior to sample collection. All other bottles, graduated cylinders and towers and bases should be detergent washed, acid rinsed and DI rinsed prior to their use in the field. Triple rinse metal utensils with DI water **do not use Acid on metal utensils.**
- 3) DOCFF samples will be obtained from the 2 liter brown sample bottles collected with the pump and hose for filtering nutrients samples.

- 4) Be sure to thoroughly mix each sample prior to pouring by gently inverting the bottles 3 times and also thoroughly rinse the filtration tower, base, graduated cylinders and tweezers with DI water and sample water between samples.
- 5) Prior to filtering, put on nitrile gloves if used.
- 6) Using clean forceps, place a new 47 mm diameter, 0.7 μm pore sized Whatman filter on the base. Replace tower.
- 7) Place a 250 mL bottle under the tower so that the stem of the base will drain into the bottle.
- 8) Transfer at least 250 mLs of sample water or DI (for equipment blank) to the tower. If the filter will be used for chlorophyll or PP analysis, use a graduated cylinder to ensure filter volume accuracy. Remove the filtrate from under the tower before adding the Magnesium Carbonate to chlorophyll samples.
- 9) Turn the pump on and filter sample. Turn the pump off as the last of the water is pulled through the filter (Vacuum pressure of pump must be 10 in Hg. (5 psi) or less. The pressure requirement must be met whether all four towers are in use or only a few towers are in use).
- **10**) Remove the sample container from under the tower.
- 11) Preserve each vial with sulfuric acid  $(H_2SO_4)$  to pH<2.
- 12) Cap and label the vials.
- Place vials in the protective sleeve and transfer the samples to the cooler. Surround the sample containers with ice to the bottom of the neck of the container.
- 14) Remove the tower and discard the filter or process the filter as needed if being used for analysis for other parameters.
- Collect an equipment blank (DI water run through the pump and hose and collected directly into 125 mL amber Nalgene bottle) and duplicates during odd months when conducting the QA/QC run (note this will mean filtering duplicates and equipment blanks for DOCFF on non-plankton sites to ensure we have approximately 10% QC samples for DOCFF. Please add the S1, S2 and EB samples to the surface samples of the PQACB and TQACB runs for March October).

#### **3.3.2 Filtering Procedures:**

Powderless, disposable gloves are recommended to for filtering. **CHECKLIST FOR FILTRATION UNIT:** 

#### EQUIPMENT FOR PROCESSING SAMPLES

PER CRUISE:
1 FILTRATION UNIT SETUP WITH AT LEAST 1 MAGNETIC GELMAN FILTER HOLDER, AND ONE -TWO PNC
TOWERS AND ONE CHLOROPHYLL TOWER
1 BOX OF 47-mm WHATMAN GF/F FILTERS (PP AND CHLOROPHYLL)
1 BOX OF 25-mm "MUFFLED" GLASS FIBER FILTERS FOR PNC
1 10-mL, 1 50-mL, 1 100-mL and one 250-mL GRADUATED CYLINDERS
1 (-2) ½ GALLON CONTAINERS
1-2 BROWN HDPE 2 LITER CONTAINER(S)
PER STATION:
2 CUBITAINERS
2 250-mL WHITE TAPE BOTTLES
2 <u>BLUE</u> TAPED PETRI DISHES (PNC)
2 GREEN TAPED PETRI DISHES (PP)
1 ALUMINUM FOIL SQUARE (CHLOROPHYLL)
POWDERLESS DISPOSABLE GLOVES

**MISCELLANEOUS** 

_ DUCT TAPE
 _ 3 FORCEPS
3 SHARPIE PENS
 _ 3 SQUIRT BOTTLES
 _ DI WATER
 5 RUBBER STOPPERS FOR FILTERING SETUP
ZIPLOCK BAGS FOR PETRI DISHES
1 ROLL EACH COLOR TAPE (BLUE, GREEN)

#### 1. Rinse filtration equipment:

If using gloves, they should be worn to clean bells (filtration towers) and frits (tower base) with deionized water (stored covered in a high-density polyethylene container) from the field office. Set up bell and frit for filtering. Ensure that there are catch flasks on line between the manifold and the vacuum source. Connect vacuum power pump to battery.

2. Place 2- 250 mL CBNUT-3 (NTNP-3 AT PLANKTON SITES) bottles under PP tower bases or under the Chlorophyll tower base(s).

Lift up base by the stopper and place <u>acid washed</u> and rinsed CBNUT-3 (NTNP-3 AT PLANKTON SITES) bottles in the bottom of the PP or Chlorophyll cylinder (s). Place the stem of the tower base inside the bottle necks such that the filtrate from the PP or Chlorophyll process will be collected in the 250-mL bottle. If the Chlorophyll tower(s) are used to collect the CBNUT-3 (NTNP-3 AT PLANKTON SITES) filtrate, the CBNUT-3 (NTNP-3 AT PLANKTON SITES) bottles must be removed prior to adding MgCO<sub>3</sub> to the last 25 mL of sample.

# 3. Place filter pads on filtration unit:

Transfer a 47-mm Whatman 0.7 GF/F glass fiber filter onto the bases for PP and Chlorophyll filtration or a muffled 25-mm filter for PNC.

- Use only clean forceps and grip only the filter edge.
- **Note:** be sure that PNC filters are muffled (Sec 2.1) prior to use and place them grid side (smooth side) down on the filter grate. Place PP and chlorophyll filters on the filter tower in the same direction as they come out of the box.
- Discard any filters if they are dropped or the surface is scratched.

Replace the filtration tower onto the base.

#### 4. Rinse graduated cylinders:

Mix sample thoroughly by inverting the plastic sample container vigorously minimum of three times, then rinse graduated cylinders with sample 1-2 times.

#### 5. Filter the samples:

**Note:** The volume of sample filtered will depend on amount of particulate matter/algae in the sample. Sample water should be thoroughly mixed by inverting the sample bottle a minimum of three times (remain consistent with the number of inversions used on to each sample bottle). Transfer the sample as quickly as possible from the sample containers to graduated cylinders, and then from the graduated cylinders to filtration towers, to prevent settling of contents. Time of transfer of sample to the towers from initial suspension should not exceed 30 seconds. If this time frame is exceeded, the sample must be re-suspended by placing lid back on the sample and inverting three times again.

- If filter pads are not "colored" following initial volume filtered, continue mixing, measuring and adding known volumes of sample water to each tower until the filters turn color.
- In cases where the sample is turbid, start with a small volume and add 50 mL increments of sample until the sample barely passes the filter (with pump on), or until the filter is well colored.
- All volume totals MUST be recorded on the sample tag (or label) and the WQM field sheet.

#### **Chlorophyll samples:**

- a. Fill the graduated cylinder with approximately 50-300 mL sample, and then quickly transfer the water from the graduated cylinder to the tower to prevent settling of algae within the cylinder.
  - Keep the vacuum below 10 in of Hg (5 psi).
  - Limit filtration duration to 10 minutes or less.
  - Remove filtration vacuum just prior to filter being completely dry but dry enough to ensure none of the filtered material will be lost when the filter is folded in the aluminum foil.
  - **Note:** 1. This procedure must be followed to avoid cell damage during filtration and loss of chlorophyll into the filtrate. If it will take longer than 10 minutes to filter the selected sample volume, discard filter and remaining sample in bell, rinse the filtration apparatus and start again using less sample volume.
    - 2. Because the laboratory has to have enough concentrated chlorophyll to obtain a spectrophotometric reading, the field crew may need to use more than 1 filter to achieve the desired amount of chlorophyll. The volume of sample filtered in combination with the color of the filter will determine how many filters should be used. In general, if you have filtered 300 mL–1 L of sample and have green color on the filter, you may use just that 1 filter. If you have filtered less than 300 mL of sample and have color other than green, you will need to filter 1 to 2 more filters. Be sure to filter the same volume in each of the successive filters (e.g. if the first filter processed 50 mL of sample and was brown in color, you will need to filter 2 more filters using 50 mL of sample each).
- b. Add MgCO<sub>3</sub> at the end of filtration.

Be sure to remove any CBNUT-3 (NTNP-3 AT PLANKTON SITES) samples prior to this step. Shake to re-suspend the MgCO<sub>3</sub> and add approximately 1 mL (about 5 drops) of concentrated MgCO<sub>3</sub> - Laboratory grade - (prepared in a 1 g MgCO<sub>3</sub> to 100 mL of deionized water ratio) to the last 25 mL (approximately) of sample filtered in the filtration bell. This is equivalent to less than 1 mg of MgCO<sub>3</sub> per 15 mL extract. The MgCO<sub>3</sub> may be added prior to the sample reaching 25 mL remaining, however be aware that the addition of the MgCO<sub>3</sub> too soon may lead to the filter clogging.

#### PNC, PP, DOC and CBNUT-3 (or NTNP-3) samples:

- The two magnetic towers with green tape are used for Particulate Phosphorus (PP) filtration.
- The two smaller towers with blue tape are used for the Particulate Carbon/Particulate Nitrogen (PNC) filtration.
- a. The usual volume filtered for PP is 250 mL (200 300 mL). The usual volume for PNC is 100 mL (100 150 mL). A greater or lesser volume may be filtered depending on sample turbidity; make sure to include the total volume filtered on the sample tag. PNC volumes are typically approximately 50% of the volume filtered for PP. Sample water should be thoroughly mixed by inverting the sample bottle a minimum of three times (remain consistent with the number of inversions used on each sample bottle). Transfer the sample as quickly as possible from the sample containers to graduated cylinders, and then from the graduated cylinders to filtration towers, to prevent settling of contents. Time of transfer of sample to the towers from initial suspension should not exceed 30 seconds. If this time frame is exceeded, the sample must be resuspended by placing the lid back on the sample and inverting three times again.

#### b. Open the vacuum valve slowly.

- Turn pump on and allow a small amount of sample to pass through the filter then turn the pump off. Remove tower from base and rinse and pour the filtrate out of the 250 mL sample bottle.
- -Turn pump back on and filter remaining sample into the 250mL sample bottle.
- After all the water has passed through the tower let run for a few seconds to pull off any water remaining on the filter.
- c. Close each valve after the sample filters through each tower. After filters are dry and colored, turn off pump.

# Keep the vacuum below 10 in of Hg (5 psi).

d. The 250-mL CBNUT-3 (NTNP-3 AT PLANKTON SITES) bottle needs to be filled at least half full.

#### Note: The filtrate should fill the bottle to just below the shoulder of the bottle.

- If there is not enough filtrate to fill the 250 mL CBNUT-3 (250 mL NTNP-3 AT PLANKTON SITES) bottle, more sample needs to be filtered. In such an event, the "used" filters must be removed, placed in a petri dish (only the first filter will be put in the petri dish and analyzed), a new filter must be placed on the tower, and additional sample (after mixing) must be filtered. If the filters are being clogged very quickly due to large amounts of solids in the sample, continue replacing the clogged filters and add sample water as necessary.
- Pour filtrate from the bottle to rinse the cap.
- The filled CBNUT-3 (NTNP-3 AT PLANKTON SITES) bottle receives the sample tag from the 2 L brown HDPE bottle (or ½ gallon jug). This bottle is delivered to DCLS.

Attach label and immediately surround bottle with ice to just below the cap of the bottle.

# Note: There have been some problems with filling the filtrate bottles to the proper volume. Common causes for these problems are:

- The old filter was not removed, water would not flow through, water in the tower and filter pad had to be thrown away and the process started again (filtrate was not contaminated and could be saved).
- New filter was not placed in tower, so sample water just poured into filtrate bottle. Bottle was contaminated and filtrate had to be thrown away (used filter pad was already in petri dish and could be used).

#### 6. Record the volume filtered.

Record the total volume filtered on the sample tag (or label) and the WQM field sheet.

#### 7. Package filter pad(s):

- Fold the filter in half using forceps, being careful not to touch or disturb the particulate material on the surface of the filter.
- Chlorophyll filter pads are placed folded on a square of aluminum foil.
- PP filter pads are placed in Petri dishes (may be sealed with green tape or a rubber band)
- PNC filter pads are placed in Petri dishes (may be sealed with blue tape or a rubber band)
- If multiple filter pads are used for chlorophyll analysis, they must be packaged as one sample. Either wrap all filter pads in one piece of foil or wrap all separate foil packages together with one piece of foil.

#### 8. Remove CBNUT-3 (NTNP-3 AT PLANKTON SITES) bottle from PP cylinder and cap tightly.

#### 9. Label sample.

Place a label on the foil square marked with:

- a) Station
- b) Date (mmddyyyy) and time (24 hour military schedule) of collection
- c) Depth of collection (m)
- d) Unit code
- e) Collector's initials
- f) Group code (PNC, PP, CBNUT-3 (NTNP-3 AT PLANKTON SITES), or FCHLR)
- g) Container number
- h) Volume of sample filtered (NOTE: When multiple filter pads are used, the volume recorded would be the combined total, i.e. if 3 pads are used and 100 mL filtered through each pad, the filtered volume recorded should be 300 mL). Do not use more than three filters!

#### 10. Place samples on ice.

- All filters may be placed together in one Ziplock bag if properly labeled.
- Make sure no water from the cooler touches the Ziplock bags.
- Place the 125 mL CBNUT-3 (250 mL NTNP-3 AT PLANKTON SITES) bottle in cooler and pack with ice to a level just below the bottom of the cap.
- 11. Completely rinse the empty filtration tower and base three times with deionized water prior to seating a new filter for the next sample. Make sure to remove tower from magnetic strip and rinse the underside of the filter grate, along with the stem of the tower for the towers that are used to collect filtrate. Also make sure to rinse any connections or seams in the towers, as these areas may collect sediment.
- 12. Empty all ½ gallon jugs and/or 2 L HDPE bottles.

#### A few things to remember:

- -Always gently invert the sample container before pouring any aliquot, making sure the sample is thoroughly mixed.
- Rinse all filtering towers with DI water between samples, paying special attention to seams, connections and stems.
- -Double check each tag for completeness and clarity.
- -Rinse graduated cylinders with DI water and then with sample water before processing each sample.
- -Rinse 125/250-mL bottle with filtrate and rinse cap with sample before closing.
- -Periodically the wastewater collection tank on the filtering units will require dumping. Make sure wastewater does not get into the overflow tank.
- -Make sure all paper work is complete and matches the sample tags exactly.

#### 3.3.3 QA/QC Sampling

Note: QA samples should be obtained in this sequence:

- 1. Field samples and duplicates
- 2. Filtration Blanks
- 3. Equipment Blanks & Clean Equipment Blanks

#### A. Equipment Blanks

Equipment blanks are used to ensure the sampling device has been effectively cleaned to prevent any carry-over from previous samples. Fill the sampling device with deionized water or pump deionized water through the device and transfer to sample bottles. Send the sample bottles to the laboratory for analysis. The Clean Equipment Blank should be collected once a year when the hoses are replaced and using a freshly cleaned filter rig. Follow the same steps as given for the monthly equipment blank below.

Samples to be collected using the methods as outlined in Section 3.0:

- 1) 1- 2 L brown HDPE bottle from which:
  - a) 300 mL of equipment blank water (DI water processed through the pump and hose apparatus) will be filtered through the filtration unit for a filter pad for chlorophyll analysis.
  - b) 100 mL of equipment blank water will be filtered through the filtration unit for a PNC filter pad.
  - c) 125-250 mL of equipment blank water will be filtered through the filtration unit for a PP filter pad. Keep the filtrate as CBNUT-3 (NTNP-3 AT PLANKTON SITES).
- 2) 1 half gallon or gallon whole water sample container for solids analysis (i.e. TSS, VSS, FSS). This container will be NME.

**Note:** NME group codes requiring larger volumes but including TSS, VSS and FSS may be substituted at CBP stations which are also AWQM sites when BOD<sub>5</sub> or other additional parameters are desired.

#### 2) Frequency and Sites of Collection:

a) Equipment blanks will be collected by all regions (NRO, TRO and PRO) on a **monthly basis**. Sites should be randomly selected. Equipment blanks will be collected on the same days duplicate samples are obtained. Regions collecting DOC for plankton sites need to collect DOC blanks (and duplicates) at a rate of 10%

Equipment blanks will be collected in the field during the run or at the regional offices at the end of the sampling day.

#### 3) Collection and Preservation Procedures:

- a) Clean containers at the regional office.
  - 1) Clean, acid wash with 10% HCL, and rinse 3 times with DI water the bucket used to hold the DI water. If a stainless steel bucket is used do not acid wash instead wash with detergent wash and DI rinse. Do the same for the graduated cylinders, the ½ gallon container (if it is being re-used) the 2 L HDPE bottles, the 250-mL CBNUT-3 (NTNP-3 AT PLANKTON SITES) sample bottle and the filtration unit stacks and bases.
- b) Rinse the outside of the hose and submersible pump with fresh water.

  Before placing the hose and pump into the bucket of deionized water, thoroughly rinse the outside of the submersible pump and hose to prevent possible contamination of the deionized water by ambient water/particulate matter remaining from the sampling run.
- c) Flush pump and hose equipment and filtration unit.

  Flush or rinse the pump and hose apparatus with sufficient deionized water to completely remove any ambient water remaining in the hose from sampling (the amount of DI water necessary for this step will vary depending on the length of hose used). Also rinse the filtration equipment with DI water 3 times (e.g. forceps, stacks, bases, graduated cylinders etc.)

#### d) <u>Collect and label samples.</u>

- 1) When ambient water has been completely flushed from the hose begin collecting the equipment blanks and process them as ambient samples.
- 2) Place a sample tag or label with the following information on each blank:
- Station name in river mile format
- Date and actual time of sample collection
- Unit code (automatically populated by CEDS)
- Collector's initials
- Analytical Group code
- Volume of sample filtered (PNC, PP and chlorophyll)
- The container number used for regular samples with the same group code but with a number 2 in front of it (e.g. if a region's regular container number for PNC is 4 the equipment blank for PNC will be 24).

Some regions have dedicated pieces of equipment for Chesapeake Bay runs. In instances where there is no dedicated equipment or if any pieces of equipment other than those dedicated are used, write on field sheet what pieces of equipment are used.

# e) Store and Preserve Samples.

- 1) Filtered: Fold the filters in half such that the inside halves of the filters touch and place in labeled petri dishes (PP and PNC) or aluminum foil (chlorophyll). Put the petri dishes and foil into Ziplock bags and then place them on ice such that they may not be contaminated by water from the melting ice.
- 2) Unfiltered: Samples should always be stored at  $\leq$  6 degrees Celsius by packing the sample bottles in ice up to the level of the top of the sample but below the bottom of the sample bottle caps.

# **B.** Field Duplicates

Duplicates are independent samples collected as closely as possible to the same point in space and time. They are two discrete samples taken from the same source, stored in separate containers, and analyzed independently (FS1 and FS2, as defined by the CBO). Duplicates are useful in documenting the precision of the sampling process.

# 1) Samples to be collected (using the methods as outlined in Section 3.0)

- **a)** 2 2 L brown HDPE bottles. Using the filtration methods outlined in the Virginia Tributary SOP collect from each bottle:
  - 1 (to 3) 47 mm chlorophyll pad(s)
  - 1 25 mm PNC filter pad
  - 1 47 mm PP filter
  - 1 250 mL CBNUT-3 (NTNP-3 AT PLANKTON SITES) bottle will be obtained through the PP filtration process.
- **b)** 2 half-gallon whole water sample containers for solids analysis (i.e. TSS, VSS, FSS) following the procedures in Section 3.0 above. This container will be NME. **Note:** NME group codes requiring larger volumes but including TSS, VSS and FSS may be substituted at CBP stations which are also AWQM sites when BOD<sub>5</sub> or other

parameters are desired.

# 2) Frequency and Sites of Collection:

- a) Duplicate samples will be collected by TRO and PRO on a **monthly basis** and by NRO on a **quarterly basis**. Regions collecting DOC for plankton sites need to collect DOC duplicates (and blanks) at a rate of 10%.
- b) Samples will be collected from both the surface and the bottom at the chosen site and sites will be rotated among stations and rivers. (Note: Bottom duplicates will not include a chlorophyll or DOC sample).

#### 3) Sample collection:

a) <u>Clean containers and filtration equipment at the regional office.</u>

Clean, acid wash with 10% HCL, and rinse 3 times with DI water the graduated cylinders, the 250 mL CBNUT-3 (NTNP-3 AT PLANKTON SITES) sample bottle, the ½ gallon container or 2 Liter HDPE sample bottle, and the filtration unit stacks and bases.

#### b) Rinse containers in the field.

If appropriate (e.g. no preservative is present in the bottle), rinse the containers once with sample water and discard rinse prior to filling with sample.

c) Rinse the filtration equipment with DI water 3 times.

Always rinse the filtration stacks, bases, forceps and graduated cylinders with DI water between filtering samples and duplicates.

#### d) Fill containers.

Filling of bottles may be accomplished by either of the following methods:

Option 1 - A "Y" fitting may be used such that both bottles may be filled simultaneously (FS1 and FS2 as defined by CBO). Leave approximately one inch of air space in each of the sample bottles.

Option 2 - A churn splitter may be filled with sample from pump and hose apparatus. While gently agitating the sample to assure homogenous sub-sampling fill both bottles sequentially leaving approximately one inch of air space (FS1 and FS2 as defined by CBO).

Option 3 – A large container (at least 1 gallon or enough to fill each sample container) may be rinsed with sample water and then filled with sample. After inverting the container at least three times to ensure homogenous sub-sampling, sample bottles are filled sequentially leaving approximately one inch of air space (FS1 and FS2 as defined by CBO). The collection container must be acid washed with 10% HCL, and rinsed 3 times with DI water prior to use in the field.

# e) Place filter pads on filtration unit.

Using clean forceps only, transfer for PP/Chlorophyll a 47 mm Whatman 0.7 GF/F glass fiber filter or, for PNC, a muffled 25 mm Whatman GFF or Pall Gelman A/E filter onto the base.

- f) Place one 125 mL CBNUT-3 (250 mL NTNP-3 AT PLANKTON SITES) bottle below the stack used for the PP filter pad.
- g) Filter and label samples.

Note: be sure to rinse all graduated cylinders, forceps and the filtration equipment with DI water between samples. Also rinse graduated cylinders with sample prior to use.

- 1) Filter 125-250 mL of sample through PP filter(s) saving one pad for PP analysis and the filtrate for CBNUT-3 (NTNP-3 AT PLANKTON SITES) analysis.
- 2) Filter enough sample to color the PNC filter pad.
- 3) Filter at least 300 mL of sample on up to 3 chlorophyll filters.

# h) Make sure to attach an identical sample tag or label to each pair of duplicates with the following information:

- 1) The duplicate samples will have station name in River mile format
- 2) Date and time of sample collection (note: the date and time should match the corresponding regular sample's date and time).
- 3) Depth
- 4) Unit code (automatically populated by CEDS)
- 5) Collector's initials
- 7) Group code
- 8) The container number used for regular samples with the same group code but with a number 1 in front of it (e.g. if a region's regular container number for PNC is 4 the duplicate container number for PNC will be 14).
- 9) Volume of sample filtered (PNC, PP and chlorophyll)
- 10) Spg code of CB on both sets of sample tags and in CEDS for both sets of samples.

#### 4) Preservation and Storage

- a) Filtered: Fold the filters in half such that the inside halves of the filters touch and place in labeled petri dishes (PP and PNC) or aluminum foil (chlorophyll). Put the petri dishes and foil into a Ziplock bag and then place them on ice such that they may not be contaminated by water from the melting ice.
- b) Unfiltered: Samples should always be stored at  $\leq 6$  degrees Celsius by packing the sample bottles in ice up to the level of the top of the sample but below the bottom of the sample bottle caps.

#### C. DI Source Blank and Conductance check of DI system.

- The DI Source blank sample is to be collected using the methods outlined previously. One 250 mL whole water sample will be filled directly from the DI tap in the laboratory for nutrient analysis. Request a NTNP-3 analysis so that SIO<sub>2</sub> can be monitored as an indicator for replacing the DI system filter.
- 2) Frequency and Sites of Collection:
  - a) DI source blanks will be collected **once a month** as follows:
    PRO will collect this sample on the York River Run (PYRK1)
    TRO will collect this sample on the Elizabeth River run (TELRIB)
    NRO will collect this sample on the Rappahannock Run (NCB)
  - b) They will be collected at the regional office.
  - c) All regions will routinely sample their DI system for conductance as this is a good indicator of filter problems.

#### 3) Collection and Preservation Procedures:

#### Collect, preserve and label samples:

- 1) Fill the containers with DI water straight from the tap.
- 2) Place a sample tag or label with the following information on the blank:
  - a) Station name in the river mile format
  - b) Date and time of sample collection
  - c) Unit code (automatically populated by CEDS)
  - d) Collector's initials
  - e) Analytical Group code
  - f) The container number used for regular samples with the same group code but with a number 4 in front of it (e.g. if a region's regular container number for NTNP-3 is 1 the equipment blank for PNC will be 41).

#### 4) Store and Preserve Samples

Samples should always be stored at  $\leq$  6 degrees Celsius by packing the sample bottles in ice up to the level of the top of the sample but below the bottom of the bottle caps.

#### D. PNC Dry Filter Blanks (PNCDF)

Two dry "muffled" filter blanks will be sent to DCLS after each sampling run. These filters will be used as a correction factor for PNC analyses.

#### Collect, preserve and label samples:

- 1. With clean forceps place the previously muffled dry filter into a petri dish sealed with blue tape.
- 2. Place a sample tag or label with the following information on the blank:
  - a) Station will be the last station in which PNC filters are obtained in river mile format.
  - b) Date and time of sample collection
  - c) Unit code (automatically populated by CEDS).
  - d) Collector's initials
  - e) Analytical Group code (PNCDF)
  - f) The container number used for regular samples with the same group code but with a number 3 in front of it (e.g. if a region's regular container number for PNC is 5 the dry filter blank for PNC will be 35).
- 3. Place the Petri dish into a Ziplock bag and send with samples to DCLS.

#### E. PP Dry Filter Blanks (PPDF)

Two dry filter blanks will be sent to DCLS after each sampling run. These filters will be used as a check for possible factory/field contamination of filters while still in the box.

#### Collect, preserve and label samples:

- 1. With clean forceps place the dry filter into a petri dish sealed with green tape.
- 2. Place a sample tag or label with the following information on the blank:
  - a) Station will be the last station in which PP filters are obtained in River mile format.
  - b) Date and time of sample collection
  - c) Unit code (automatically populated by CEDS).

- d) Collector's initials
- e) Group code: PPDF
- f) The container number used for regular samples with the same group code but with a number 3 in front of it (e.g. if a region's regular container number for PP is 6 the dry filter blank for PP will be 36).
- 3. Place the Petri dish into a Ziplock bag and send with samples to DCLS.

#### F. Filtration Blanks

Filtration blanks are used to ensure the filter towers and bases have been effectively cleaned to prevent any carry-over from previous samples. Rinse the graduated cylinders, towers and bases with DI water as is done between samples. Replace the filter bases and towers and transfer the usual amount of deionized water as you would for a routine sample. Package and ice the samples as is done with the regular samples. The CEDS blank/dup designation for these samples is FDI for Filtered DI blank.

#### 1) Samples to be collected using the methods as outlined in Section 3.0:

- a) 300 mL of DI water will be filtered through the filtration unit for a filter pad for chlorophyll analysis.
- b) 50-100 mL of DI water will be filtered through the filtration unit for a PNC filter pad.
- c) 100-250 mL of DI water will be filtered through the filtration unit for a PP filter pad. Keep the filtrate as CBNUT-3 (NTNP-3 AT PLANKTON SITES).

#### 2) Frequency and Sites of Collection:

- a) Filtration blanks are not currently retained as part of the regular QA/QC process.
- b) In the event an equipment blank is not possible a filtration blank may be substituted.

#### 3) Collection and Preservation Procedures:

See Section 3.3.3, part A, step 3 above.

#### 4.0 POST CRUISE ACTIVITIES

#### 4.1 SAMPLE CUSTODY AND HANDLING

- 1. Drain and repack ice coolers.
  - Using the drain plug on the coolers, remove any water from the coolers.
  - Repack samples to the bottom of their caps with ice.
  - Check to make sure the sample tags and laboratory sheets (when used) and WQM Data sheets match and are completely filled out.
- 2. Place coolers where they will be picked up by the DCLS courier.
  - There may be occasions when a DCLS courier is unable to pick up the coolers.

Should such a situation arise, deliver the samples to DCLS:

- DCLS is located at 600 North 5<sup>th</sup> St. in Richmond, VA.
- Parking is available in the loading dock (4<sup>th</sup> Street between Leigh and Jackson) or on 4th Street adjacent to the loading dock.

If the overhead doors to the loading dock are closed, go to door on the side of the overhead doors and use the intercom to contact the loading dock security officer. If the overhead doors to the loading dock are open, you will find the loading dock security officer behind the glass window of the loading dock office. The security officer will assist you checking the samples into the lab.

#### 4.2 Multiprobe post cruise calibration checks

**Note:** Perform a post cruise calibration check before cleaning/servicing the sensors following the agency WQM SOP. When checking the system for drift, it is extremely important that the room temperature, sonde temperature, deionized water temperature, and all standard solutions are at thermal equilibrium. If thermal equilibrium is not reached in a reasonable amount of time or the observed values are outside the QC criteria, an additional post cruise calibration check should be conducted the next morning. When a post cruise calibration check is necessary the following morning, send those results to CBO with the field data sheets.

#### 4.2.1. QA/QC Criteria

#### **Dissolved Oxygen**

- 1. Make sure the multiprobe returns to ambient temperature prior to conducting the calibration check.
- 2. Compare the instrument and chart values.

Compare the Saturated DO values from the chart and the instrument values as recorded in the logbook. If the difference between the two is less than 0.3 mg/L the instrument is in calibration. If the difference between the Saturated DO value and the instrument indicates that the instrument is not in calibration, check again the next morning to make sure that the temperature was properly equilibrated (send these values to CBO). If the difference is still greater than or equal to 0.3 mg/L the unit should not be used to collect additional measurements until more extensive cleaning/maintenance is conducted and the instrument calibrates well.

#### **Specific Conductance**

1. Compare the instrument and standard values.

Compare the conductance from the instrument as recorded in the logbook to the standard value. If the standard value and the instrument value are not in good agreement, perform an additional post cruise calibration check the following morning to ensure the instrument was properly equilibrated (send these values to CBO). Record the data on the logsheet. Compare the displayed value to the standard value and calculate the difference. If the difference is less than +/- 5% of the standard used, then the instrument is in calibration. If the instrument is not in calibration, check again the next morning to make sure that the temperature was properly equilibrated. If the difference is still out specification, the data is suspect and should not be entered into CEDS. Additionally, the multiprobe should not be used for that parameter until it has an extensive cleaning/maintenance.

If the data is determined to be suspect, and the data have been entered into CEDs, it will be removed from the database. The multiprobe should not be used until more extensive cleaning/maintenance is conducted and the instrument calibrates well.

#### pН

1. Compare instrument value and the standard value.

Compare the pH value displayed on the instrument to the standard value. If the difference between the standard and the instrument is  $\leq 0.2$  SU, then the instrument is in calibration. If the difference between the pre-calibration and post-calibration indicates that the instrument is not in calibration, check again the next morning to make sure that the temperature was properly equilibrated (send these values to CBO). If the difference exceeds 0.2 SU the data will be deleted from the database. That multiprobe should not be used until more extensive cleaning/maintenance is conducted and the instrument calibrates well.

#### 4.3 ACID WASH REUSABLE BOTTLES

All reusable sample bottles and coolers should be returned from DCLS to the regions requesting them via the courier on a regular basis. Contact Cindy Johnson at CO if there is a problem in getting coolers or sample bottles back from DCLS. All 125 mL CBNUT-3, 250 mL NTNP-3 (for plankton sites) and 2 L brown bottles need to be detergent washed and acid washed between runs. This step may be performed as a pre-cruise preparation or in the afternoon as a post-cruise activity.

Rinse reusable bottles with tap water, strip with 10% HCL and rinse 3 times with DI. Reusable bottles may consist of 125 mL CBNUT-3 (250 mL for NTNP-3 AT PLANKTON SITES) bottles, large chlorophyll-a bottles (brown 1 L bottles) and their caps. Replace the caps to seal bottles until their use.

#### 4.4 EQUIPMENT MAINTENANCE

#### **4.4.1. HOSES**

1. Because of the danger of contamination by bacteria and other particulate matter, the hoses used

in the Chesapeake Bay's Virginia Tributary Monitoring Program (VTMP) are to be replaced annually.

Hoses consisting of ¾" ID by 1 1/8 inch OD clear vinyl tubing are currently being used by the VTMP.

2. To deter bacterial and algal growth in the tubing between purchase and replacement, the following cleaning should be conducted:

#### AT THE END OF EACH SAMPLING RUN:

Rinse the hoses with 5 gallons of tap water and completely drain.

#### **ONCE PER MONTH:**

- a. Pump 5 gallons of a 5-10 % solution of white vinegar mixed with tap water through the hose and pump apparatus (a 5 % solution consists of 1 quart of vinegar mixed with 4 ¾ gallons of water).
- b. Rinse with 5 gallons of pure tap water and drain.

#### 4.4.2 SECCHI DISK

Once per month clean the Secchi Disk with detergent.

#### **4.4.3 LI-COR SENSOR MAINTENANCE:**

#### AT THE END OF EACH SAMPLING RUN:

Rinse the underwater sensor and cable with tap or DI water.

#### SIX MONTHS AFTER MANUFACTURE'S RECALIBRATION:

The air and water sensors should be compared to each other at the regional office. Program the air multiplication factor into the display unit for the underwater sensor following the instructions on p.24 and obtain a simultaneous reading from the air and water sensors. The air and water sensors should be within 5% of each other. If not, try to determine which sensor is not functioning properly by comparing the air and water sensors to another unit's sensors. Non-functioning sensors should be sent back to the manufacturer for repair.

#### **EVERY YEAR (max. 2 years if necessary):**

Return the sensors to the manufacturer (LI-COR) for calibration. Send a copy of the calibration drift report to CBO upon receipt.

#### 4.5 ELECTRONIC DATA TRANSFER (EDT) OF SAMPLE INFORMATION

Note: Secchi depth, weather code and tide stage for each station should be entered in WQM only once with the surface depth profile information.

#### 4.5.1 Regular run information

- 1. CBM WQM data sheets are filled out at each station.
- 2. Send the WQM data sheet information via WQM to DCLS prior to 9:00 am on the day following the sampling cruise.
  - Record each sample sent to DCLS at each station and depth profile
  - Check data

- 3. Scan or make copies of the WQM field sheets and send them along with the Li-Cor and Field Summary sheets to CBO after each cruise May through September and at the end of the month the remainder of the year (CBO will do QC on the data entry).
- 4. If technical problems arise during the data shipment and the 9:00 am deadline will not be met call Cindy Johnson (804-659-2653) or another appropriate OIS/WQA staff member (see call list in Appendix D). If the problem cannot be resolved, fax the WQM field sheets to DCLS Sample Support Services (804-786-4270) and call Cindy Johnson at CO (804-659-2653)

#### 4.5.2 QA/QC run information

Entering QA/QC data into the Oracle database requires special steps not normally performed during a regular run. See Appendix D for details.

#### 4.6 CORRECTIVE ACTION REQUEST

The corrective action request form (CAR; Appendix A) is used to document problems that might affect data accuracy, data precision, or sampling efficiency and to recommend changes for correction. CAR forms may originate in regions, headquarters, or the labs. The main reason to use a CAR is the need to permanently change procedures. CAR's may occur because current procedures may be causing sample contamination, are in need of clarification, or are inconsistent with new research on best practices.

In order for the corrective action plan to work, all personnel associated with the program must report all suspected abnormalities. This is especially important to field personnel because identification and correction of problems in sample collection and handling is essential for an effective program. The general steps in the CAR process are:

- Identify the problem.
- List possible causes (if known).
- Note the date the problem was identified.
- Identify samples or field data that may be invalid as a result of the problem.
- Make recommendations for corrective action (if possible).

# APPENDIX A LAB SHEETS AND FORMS

Revised 04/02/2020

#### FIELD SUMMARY

River:		Date:		_
Field Chief:				
Field Crew:				
Weather:				
Station	Time	Tide	Comments	/Problems/Unusual Events
Calibration Check (Perfo	ormed after sa	mpling)		AV.
Sonde #				Notes:
Date/Time		•		
Conductivity	S.V.			
	I.V.			
рН	S.V.			
	I.V.			
DO	S.V.			
	I.V.			
S.V. = Standard Value I.V. = Instrument Value				
Signature:		]	Place <b>the form</b> in the Region	nal Sharepoint folder by the end of the month

 VIRGINIA DEQ
 Collector
 STATION

 Program
 CB
 LDS
 2-APP001.53 (TF5.4)
 Buoy 8

 Sampled
 Lat 37° 18' \_\_\_"
 Long -77° 17' \_\_"
 \_"

 Target
 Lat 37° 18' 44.64"
 Long -77° 17' 28.78"

D			FIELD DATA		
E P T H(m)	TEMP(°C) 00010	pH (SU) 00400	D.O.PROBE (mg/l) 00299	COND. (uMHOS/CM) 00094	SALINITY (ppt) 00096
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					

Sample Date/_/	Sample Time
Observed Total Depth	(meters)
Secchi Depth (m)	
Weather	Tide
Codes: 1 – Cloudy	Codes: 1 – High
2 – Precipitation	2-Low
3 – Clear	3 – Flood
4-Fog	4 - Ebb

#### **Containers to Collect**

LAYER	VOLUME FILTERED	GROUP CODE	CONTAINER NUMBER
S U		NME12	1
R F		CBNUT-3	2
A C		FCMFENT 4	3
Е		PNC	5
Filt.Time:		PP	6
Init:		FCHLR	7
В		NME7	1
T T		CBNUT	2
O M		PNC	5
Filt. Time:		PP	6

Virginia
Department
of
Environmental
Quality

QualityDCLS LAB USE ONLY STATION ID DATE COLLECTED SURVEY DEPTH TIME COLLECTED PROG. PRIORITY CODE **GROUP CODE** CONTAINER # UNIT CODE REGION CODE COLLECTOR FLOW SEVERITY FIELD pH SPECIAL STUDY NUMBER %FRB WEATHER TIDE SECCHI DEPTH (m) 00041 00067 01351 00078 00116 00002 00400 BAROMETER PRESSURE RESIDUAL CHLORINE FLOW RATE COLLECTION SPAN # OF ALIQUOTS AIR TEMP. (C°) 50060 00025 00061 00020 SPWL SWL HOURS YIELD G W TIS (NUM) SPECIES (NUM) SAMPLE NO. TIS (ALPHA) SPECIES (ALPHA) ı D S 7499 7499 8400 8400 5 IND/SAMPLE WEIGHT (LBS.) S SEX LENGTH (INCHES) LC/H U Е 8161 8400 8401 00024 00023 8 LATTITUDE DE FIELD DATA DO PROBE (mg/L) РΤ TEMP ° C COND. (µ MHOS/CM) SALINITY (ppt) LONGITUDE H (m) 00299 00010 00094 00096 OTHER 11 13 COUNTY 15 COM-MENTS 19 21

#### DEPARTMENT OF ENVIRONMENTAL QUALITY

#### Li-Cor LIGHT ATTENUATION York River

Collected By	Date:
Light attenuation measurements are take	en at one half meter intervals until a reading of 10 uE is reached.

**STATION:** <u>**RET4.3**</u> 8-YRK031.39

TIME:

DEPTH (M)	AIR/WATER	DEPTH (M)	AIR/WATER
SURFACE	/	6.0	/
0.5	/	6.5	/
1.0	/	7.0	/
1.5	/	7.5	/
2.0	/	8.0	/
2.5	/	8.5	/
3.0	/	9.0	/
3.5	/	9.5	/
4.0	/	10	/
4.5	/	10.5	/
5.0	/	11.0	/

## DEPARTMENT OF ENVIRONMENTAL QUALITY CORRECTIVE ACTION REQUEST FORM (CAR)

Section I - Completed by originator
Date: Submitted By: Region:
A. Nature of Problem:
B. Possible cause (if known):
C. Date problem identified:
D. Samples that may be invalid:
E. Recommended Corrective Action (optional):

#### D.E.Q. CAR continued

## Section II - Completed by Regional Technical Services Supervisor

A. Recommended Corrective Action:
Technical Services Supervisor, Signature:
Date:
Section III - Completed by CBO Monitoring Project Coordinator
A. Recommended Corrective Action:
B. Follow up action required: YES / NO
C. Implementation will begin on:
CBO Project Coordinator Signature:
Date:
Section IV - Completed by Headquarters QA/QC (optional)
A. Recommendations / Comments:
QA/QC Signature:
Date:

Multiprobe Cal Log Sheet Sonde Make/Model:

Sonde S/N:

						DO		Specific Co	onductivity	
Cal Type	Date/Time	Initial and Run ID	Temp C	BP (mmHg)	Chart DO	Meter DO	Cal DO	Cond Std. (uS/cm)	Cond Init/Cal	pH 7 Init/Cal
Pre			<u> </u>							
Post						'		4		
Comments					DO QA:	:		Cond QA:		pH QA:
Pre										
Post								4		
Comments:	,				DO QA:	:		Cond QA		pH QA:
Pre					<b>_</b>	<u> </u> '			<u> </u>	↓
Post						<u> </u>		<u> </u>		
Comments:					DO QA:	:		Cond QA:		pH QA:
Pre										_
Post										
Comments:	;				DO QA:	<u>.</u> :		Cond QA:		pH QA:
Pre						<u> </u>			<u> </u>	<del> </del>
Post										
<b>Comments:</b>		_		_	DO QA:	:		Cond QA:		pH QA:

DO QA: YSI ODO Gain 0.75 to 1.25, Cond QA: YSI Cell Constant 4.55 to 5.45 pH QA: pH 7: -50 to 50 mV, pH 4: 130 to 230 mV, pH

## **Record of Multiprobe Maintenance Procedures**

Multiprobe #	
--------------	--

Date	Procedure	Comments

## **CBP Tributary Site Visit Summary**

rsonnel:			
n:			
sit by:			
re cruise Procedures:			
<u>-</u>	Yes No	<u>N</u>	<u>I/A</u>
1. Precalibration normal and in accordance with SOP.	Ц	Ц	Ц
2. All expected parameters calibrated.			
3. Expiration date not exceeded on pH buffer, 1.0 Molar stock solution less than 1 year old.			
4. Used fresh Standards to calibrate conductivity.		H	H
5. Instrument operation good, in accordance with SOP.		П	Ħ
6. Regional office maintains calibration/maintenance logbook.			
<ul><li>A2. Sample Container and Filtration Equipment Preparation:</li><li>1. Filtration equipment (towers, bases, 250 mL bottles, graduated</li></ul>	Yes	<u>No</u>	<u>N/A</u>
cylinders and 2 L HDPE brown bottles ) washed with liquinox			
detergent, acid washed, and rinsed 3 times with DI water 2. Filtration utensils/new sample bottles DI water rinsed		Ш	
3 times.			
A3. Li-cor Unit Preparation:	Yes	No	N/A
1. Sensors washed with mild detergent such as Liquinox®	105	110	<u>11/11</u>
prior to cruise			
2. Averaging time set to 5 seconds			
3. Multipliers entered correctly			
4. Calibration log maintained			
A4. NIST Thermistor Check	Yes	<u>No</u>	<u>N/A</u>
1. Temperature difference between probe and NIST certified thermistor at 25-30 °C is +/- 0.5 °C probe value :			
Nist certified thermistor value :			
2. Temperature difference between probe and NIST certified thermistor at 4°C is +/- 0.5 °C			
probe value :			
Nist certified thermistor value :			

## **B. Field Collection Procedures:**

<ol> <li>B1. Water Sample Collection:         <ol> <li>Stations sampled according to original schedule.</li> <li>All stations sampled during daylight hours.</li> <li>Obtained samples 1 m above bottom sediment/1 m below surface.</li> <li>Samples collected via pump and hose apparatus.</li> <li>Pump adequately cleared/flushed.</li> <li>Sample bottles sample rinsed prior to sample collection.</li> <li>Sufficient sample volume collected for all parameters.</li> </ol> </li> <li>Samples properly labeled using ink.</li> <li>Samples iced immediately/preserved according to SOP.</li> </ol>	Yes		
<b>B2.</b> Multiprobe Procedures:	<u>Yes</u>	<u>No</u>	N/A
<ol> <li>Readings in 1 m increments from 1 m above bottom sediment (raised to 1st whole odd interval) to 1 m below surface.</li> <li>Multiprobe readings stabilized prior to recording information on data sheets.</li> </ol>			
<ol> <li>B4. General Filtration Procedures:</li> <li>DI water rinsed filtration towers, bases, tweezers, graduated cylinders prior to use /between samples according to SOP.</li> <li>Graduated cylinders rinsed with sample prior to use.</li> <li>Filters stored properly prior to use.</li> <li>Only clean forceps used for filter transferals.</li> <li>Filters handled properly.</li> <li>Sample re-suspended by shaking for filtration.</li> <li>Volumes filtered recorded on tags and data sheets.</li> <li>All sample filters kept in Ziplock storage bags, iced immediately after collection.</li> </ol>	Yes	No	N/A
<ol> <li>B5. Chlorophyll Filtration Procedures:</li> <li>Vacuum pressure during filtration between 10 in Hg, 5 psi.</li> <li>Sufficient sample volume filtered to color filter pad, if less than 300 mL of sample filtered through the first pad, filtered one/ two more filter pads to obtain a total of at least 150 mL of sample filtered on each pad.</li> <li>Filtration duration limited to 10 minutes or less.</li> <li>Added MgCO3 to last 10-25 mLs of sample being filtered.</li> <li>Removed filtration vacuum prior to filter being completely dry/but dry enough to ensure no sample losses during filter folding.</li> <li>Wrapped filters containing chlorophyll samples in aluminum foil/ immediately iced them according to SOP.</li> </ol>	Yes	No	N/A
<ul> <li>B6. Nutrient Filtration Procedures:</li> <li>1. Vacuum pressure kept 10 in Hg, 5 psi or below.</li> <li>2. Muffled PNC filters prior to use, placed filters grid side down according to SOP.</li> <li>3. Filtered sufficient volume of sample to obtain color.</li> </ul>	Yes	No	<u>N/A</u>

<ol> <li>Miscellaneous:</li> <li>Secchi depth measured during daylight hours on shady side of vessel without using sunglasses.</li> <li>Duplicate samples obtained according to SOP.</li> <li>Equipment blank samples obtained according to SOP.</li> <li>Any problem noted but not specifically listed above.</li> <li>Is PMTF required?</li> </ol> Comments:	Yes	No	<u>N/A</u>
C. Phytoplankton and Productivity Sample Collection	ns		
C1. Light Attenuation Procedures:	Yes	<u>No</u>	N/A
1. Initial readings obtained with deck sensor/ 0.1 meter with water sensor, subsequent readings in 0.5 meter increments.			
2. Sensor position minimized surface reflection.			
3. Deck/water profiles continued to depth value of 10 micro			
Einsteins or less. 4. Readings were obtained after waiting 15 seconds.		$\mathbb{H}$	
4. Readings were obtained after waiting 15 seconds.			
C2. Photic zone	Yes	<u>No</u>	N/A
1. Photic zone composites consisted of 5 equal subsamples			
determined by multiplying the Secchi depth, depth by 3.5 (or from chart based on Secchi depth) and dividing that by 5			
2. The hose was cleared adequately between depths			
3. The carboys were mixed thoroughly prior to pouring the samples			
<ul><li>4. Bottles were not overfilled</li><li>5. The water temperature was clearly written on the tops of the</li></ul>		Ш	
productivity samples			
6. Samples were labeled properly			
C3. Sample delivery	Yes	No	NI/A
1. Delivery times were confirmed with ODU ahead of time			
2. Samples were properly iced and delivered same day to ODU			
D. Post Cruise Procedures:			
D. Tost Cruise Procedures.			
D1. Field Personnel Post Cruise Procedures:	<u>Yes</u>	<u>No</u>	N/A
<ol> <li>Sonde post cruise calibration check conducted according to SOP/normal.</li> </ol>			
2. Hose cleaning/draining procedures completed according to SOP.	H	H	H
D2 Data Entry	Yes	<u>No</u>	$\overline{N/A}$
1. Three months of field sheets were selected randomly by CO			
personnel to review.  2. Percent errors found equaled 10 percent or less	$\mathbb{H}$	H	H
a. Total number errors found =		Ш	
b. Total number data points reviewed =	_		
Percent error = step a/step b*100% =			

#### VIRGINIA TRIBUTARY MONITORING PROGRAM PROCEDURE MODIFICTION TRACKING FORM

This form is used to document modifications made to the Virginia tributary Monitoring Program's procedures or methods. A detailed method description including the proposed modification should be completed prior to submittal to DEQ's Chesapeake Bay Program at the Central office.

DATE SUBMITTED		DATE APPROVED	
REQUESTOR NAME		ORGANIZATION	
NEWLY PROPOSED MODIFIC	CATION [ ]	FIELD APPROVED MODIFICATION [ ]	
APPROVED BY:		DATE:	
TYPE OF PROCEDURE/METHOD	SAMPLING [ ] ANALYTICA OTHER [ ] SPECIFY:	L[] FIELD MEASUREMENT[]	
DURATION	PERMANENT [ ] EFFECTI TEMPORARY [ ] START D END DA		
PROCEDURE/METHOD DESCRIPTION			
MODIFICATION DESCRIPTION			
JUSTIFICATION FOR MODIFICATION			
ANALYTICAL PARAMETERS THAT MAY BE AFFECTED BY THIS CHANGE			
AFFECTED QA PLAN(S) (INCLUDE TITLE, REVISION AND DATE)			
PMTF COMPLETED BY			

\_TITLE:\_\_\_\_SI

DATE:\_\_\_\_

CBO REVIEW/APPROVAL:

GNATURE:\_\_\_\_

## Field Filtration Log

RO	RO Station		Station	Station Da	Date	Sample Type	Depth (m)	Time Collected	Time Filtered	Volume Filtered (mL)		
							Chl-a	PNC	PP			
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			R/S1									
			R/S1	1								
			R/S1									
			R/S1	1								
			R/S1									
			S2	1								
			S2									
			EB	0								

## LI-COR SENSOR TRACKING LOG

#### **Sensor serial number:**

#### **Purchase date:**

Multiplion	Medium	Calibration	Field Usation		Comments
Mulupher	air/water	Drift	Start Date	Stop Date	
	Multiplier			Multiplier	Multiplier

## **APPENDIX B**

## **Saturated DO Table**

and

**Primary Productivity Sample Increment Chart** 

Revised 04/08/2002

## Saturated DO Table

Temp				O <sub>2</sub>	concentrat	tions in mg	L			
in <sup>O</sup> C	0	.1	.2	.3	.4	.5	.6	.7	.8	.9
5	12.75	12.71	12.68	12.65	12.61	12.58	12.55	12.52	12.48	12.45
6	12.42	12.39	12.36	12.32	12.29	12.26	12.23	12.20	12.17	12.14
7	12.11	12.08	12.05	12.02	11.99	11.96	11.93	11.90	11.87	11.84
8	11.81	11.78	11.758	11.72	11.69	11.67	11.64	11.61	11.58	11.55
9	11.53	11.50	11.47	11.44	11.42	11.39	11.36	11.33	11.31	11.28
10	11.25	11.23	11.20	11.18	11.15	11.12	11.10	11.07	11.05	11.02
11	10.99	10.97	10.94	10.92	10.89	10.87	10.84	10.82	10.79	10.77
12	10.75	10.72	10.70	10.67	10.65	10.63	10.60	10.58	10.55	10.53
13	10.51	10.48	10.46	10.44	10.41	10.39	10.37	10.35	10.32	10.30
14	10.28	10.26	10.23	10.21	10.19	10.17	10.15	10.12	10.10	10.08
15	10.06	10.04	10.02	9.99	9.97	9.95	9.93	9.91	9.89	9.87
16	9.85	9.83	9.81	9.79	9.76	9.74	9.72	9.70	9.68	9.66
17	9.64	9.62	9.60	9.58	9.56	9.54	9.53	9.51	9.49	9.47
18	9.45	9.43	9.41	9.39	9.37	9.35	9.33	9.31	9.30	9.28
19	9.26	9.24	9.22	9.20	9.19	9.17	9.15	9.13	9.11	9.09
20	9.08	9.06	9.04	9.02	9.01	8.99	8.97	8.95	8.94	8.92
21	8.90	8.88	8.87	8.85	8.83	8.82	8.80	8.78	8.76	8.75
22	8.73	8.71	8.70	8.68	8.66	8.65	8.63	8.62	8.60	8.58
23	8.57	8.55	8.53	8.52	8.50	8.49	8.47	8.46	8.44	8.42
24	8.41	8.39	8.38	8.36	8.35	8.33	8.32	8.30	8.28	8.27
25	8.25	8.24	8.22	8.21	8.19	8.18	8.16	8.15	8.14	8.12
26	8.11	8.09	8.08	8.06	8.05	8.03	8.02	8.00	7.99	7.98
27	7.96	7.95	7.93	7.92	7.90	7.89	7.88	7.86	7.85	7.83
28	7.82	7.81	7.79	7.78	7.77	7.75	7.74	7.73	7.71	7.70
29	7.69	7.67	7.66	7.65	7.63	7.62	7.61	7.59	7.58	7.57
30	7.55	7.54		7.51	7.50	7.49		7.46 mm Hg	7.45	7.44
mm Hg.	Corr.F		mm Hg.	Corr.Facto			Corr.Factor		Co	rr.Factor
775	1.02		750-746	0.987		5-721	0.953	700-6		
770-766	1.014	1	745-741	0.98		-716	0.947	695-6	91 0.9	914
765-761	1.007	7	740-736	0.973	715	5-711	0.94	690-6	86 0.9	907
760-756	1.0	,	735-731	0.967	710	-706	0.934	685-6	81 0.9	90
755-751	0.993	3	730-726	0.96	705	5-701	0.927	680-6	76 0.3	893

## Primary Productivity Sampling Increment Charts

#### A. TOP SAMPLE

Secchi Depth (m)	Sampling Start Depth (m)	Sampling Increment (m) 5X
0.1	0.35	.07
0.2	0.7	.14
0.3	1.05	.21
0.4	1.4	.28
0.5	1.75	.35
0.6	2.1	.42
0.7	2.45	.49
0.8	2.8	.56
0.9	3.15	.63
1.0	3.5	0.7
1.2	4.2	0.84
1.4	4.9	0.98
1.6	5.6	1.12
1.8	6.3	1.26
2.0	7.0	1.4

# APPENDIX C COMMONLY USED GROUP CODES AND CONTAINER NUMBERS

Revised 4/1/2015

Depth	Group Code	Container Number
	FCMFECQENT	1
	TNUTL (at TF3.1F only)	2
	NME7	3
S	CBNUT-3	4
	PNC	5
	PP	6
	FCHLR	7
	CBNUT-3	4
В	PNC	5
	PP	6
	NME7	8

#### PRO

Depth	Group Code	Container Number
	NME7	1
	CBNUT-3 (NTNP-3 at	2
	plankton sites)	
C C	FCMFECQENT	3
S	PNC	5
	PP	6
	FCHLR	7
	DOCFF (Plankton sites only)	8
	NME7	1
В	CBNUT-3 (NTNP-3 at	2
	plankton sites)	
	PNC	5
	PP	6

#### TRO

Depth	Group Code	Container Number
	NME7	1
	CBNUT-3 (NTNP-3 at	2
	plankton sites)	
C	FCMFECQENT	3
S	PNC	4
	PP	5
	FCHLR	6
	DOCFF (at plankton sites)	7
	NME7	1
В	CBNUT-3 (NTNP-3 at	2
	plankton sites)	
	PNC	4
	PP	5

<sup>\*</sup> Group codes collected on stations that are sampled for the AQ program.

## **APPENDIX D**

## **ENTERING QA/QC INFORMATION**

## INTO WQM and

## CALL LIST FOR SAMPLE RELATED ISSUES

Revised 06/27/2023

#### QA/QC Checklist for WQM

## (Note: If problems occur during data entry use the call list on the next page to get help with the issue).

A. If a run does not exist, create a QACB Run (e.g. PQACB for PRO; NQACB for NRO and TQACB for TRO). The QA run should contain a valid station (QA is a valid station in CEDS), a default depth for the bottom stations, containers 11-19 for S2 samples (needed for both surface and bottom depths) and containers 21-29 for equipment blanks at depth 0.

B. If the QA run has been established use the Merge QA/QC button in the Run screen to schedule your QC samples.

- 1. Click on Schedule Run button and enter date the Run will be conducted.
- 2. Go to the station where the QAQC will be collected click on the Merge QA/QC Run button. In the popup enter the name of the QAQC run to be merged and save.
- 3. Change Blank/Dup designation for the station where duplicates will be collected from R to S1 for containers 1-9 (the Blank/Dup designations may also be changed in the Field data screen).
- 4. Change bottom depth to correspond with S1 bottom depths.
- 5. Change station order number to correspond with the station order number in the routine run.
- 6. Make sure containers 11-19 are coded as S2.
- 7. Make sure containers 21-29 are coded as EB.

#### C. In Field Data:

1. Enter sample time and field data. If you have not already done so, Click on the Samples button, the Edit button for each container collected and change Blank/Dup from R to S1 for containers 1-9 at the station chosen for QA/QC.

Entering QA/QC data into WQM: April 4, 2019

Note: Advance scheduling of sampling runs must be completed by the 25<sup>th</sup> day of the month prior to the month of sample collection. A complete tutorial can be found at the following site:

#### A. Collection of QA/QC samples:

The types and frequency of collection of QA/QC samples is described in each individual program's SOP.

#### **B. QA/QC Run IDs:**

QA/QC Run IDs consists of the first letter of the region conducting the sampling followed by the letters QA and the 2-letter program code under which the samples are collected (e.g. TQAAQ for the Tidewater regional QA run for the Ambient Monitoring Program).

## C. Blank/Dup designations (Note: all QA/QC samples are stored under the QA/QC Run ID except FB and S1).

CRM - Certified Reference Material	C - Composite Sample
CB - Container Blank	EB - Equipment Blanks
FB - Filter Blank	FDI-Filter apparatus equipment blank
H - Horizontally integrated composite sample	HV - Horizontally and Vertically integrated sample
M - Multiple Samples	R - Regular Sample (default designation)
RB - Reagent Blank	<b>S1</b> - First subsample of a field split sample (these data
-	are stored in the regular run id)
S2 - Second subsample of a field split sample (these	SRM - Standard Reference Material
data are stored in the QA/QC run ids)	
TB - Trip Blank	V - Vertically integrated composite sample

#### **D.** Container number designations:

1 - 9	Regular sample containers and/or S1 sample containers
11 - 19	S2 sample containers (note the ones place of a S2 container is the same as the
	corresponding S1 container e.g. if S1 for PNC is container number 5 then S2 for PNC is
	container number 15)
21-29	Equipment Blanks (the ones place for equipment blanks also correspond to the S1
	containers for the same sample types e.g. S1 for PNC is 5 then the EB for PNC is 25).
31-39	Filter Blank (the ones digit must correspond to the S1 or R sample container for the
	group code using that type of filter e.g. PNC)
41-43	FDI Blanks
51-53	Reagent Blanks

#### E. Lab Proc Code designations:

**D** - indicates to the lab to perform Laboratory splits

**DM** - indicates to the lab they should perform a lab split and a matrix spike

M - indicates to the lab they should perform a matrix spike

#### CALL LIST FOR SAMPLE RELATED ISSUES

04/15/2022

This is a list of persons, listed in order of priority, to call for help to the listed problems.

#### **EMERGENCY LABORATORY SERVICES (all area code 804)**

After Hours Emergency Services Officer 804-335-4617 (Cell Phone)

Shane Wyatt- DCLS Director, Laboratory Operations & DEQ Coordinator

648-4480 x152 371-7973 (fax) Shane. Wyatt@dgs.virginia.gov

#### **ROUTINE SAMPLE DELIVERY PROBLEMS (all area code 804)**

Cindy Johnson	659-2653	334-7590 (C)
Roger Stewart	698-4449	370-8043 (C)

Drew Garey 698-4253

Terri Harper-DCLS 648-4480 ext. 140 Elaine Mason 648-4480 ext. 138

#### PROBLEMS SPECIFIC TO DATA TRANSFER

Cindy Johnson	659-2653*	334-7590 (C)	
Roger Stewart	698-4449*	370-8043 (C)	
Drew Garey	698-4253*		
DEQ Help Desk	698-4100		
Sai Ram Thotakura	512-9588698-45	48512-9588 maintains data transfer site.	

<sup>\*</sup> Can perform manual download of WQM data to ship to DCLS.

#### SAMPLE COLLECTION INFORMATION & SCHEDULING WITH DCLS

These numbers are provided for non-routine sample collection and scheduling. Please make certain when scheduling bacteria samples that you <u>confirm</u> that one of the following persons know when and how many samples will be arriving and what services will be requested.

Cindy Johnson 659-2653(O) 334-7590 (C)

Cindy.johnson@deq.virginia.gov

Shane Wyatt – Director, Laboratory Operations & DEQ Coordinator

648-4480 x152 641-7056 (C) FAX

Shane.Wyatt@dgs.virginia.gov

Bailey Davis - Inorganic Chemistry and Water Microbiology Group Manager Bacteria sample scheduling only

648-4480 x320 FAX

Bailey.davis@dgs.virginia.gov

#### ORDERING SAMPLE KITS AND CONTAINERS

Terri Harper, SSS Group Manager 648-4480 ext. 140 FAX 786-4270

Mattie Jones, DCLS Customer Service Support Technician 648-4480 ext. 104

#### ORDERING CLEAN METALS KITS

804-648-4480 x 354 David.Gulick@dgs.virginia.gov

#### **COURIER SERVICE**

Terri Harper, SSS Group Manager 648-4480 ext. 140 FAX 786-4270

#### **PRIORITY CODES**

Every priority code other than the standard 7 (the usual turnaround time (TAT), as listed in the catalog of services) has a cost multiplier associated with it.

Code 7 – standard TAT, listed price

Code 6 - Chain of custody, standard TAT, listed price

Code 5 - ½ standard TAT, 1.5 X listed price

Code 4 – 7 day TAT, 2 X listed price

Code 2 – Chain of Custody for samples that will likely be used for litigation, standard TAT 1.1 X listed price.

Code 1 – Emergency sample. Pricing will be determined after completion of analysis. Since this requires lab employees to work around the clock to complete the analysis, these samples must be approved by a RD or agency director.

Bear in mind that timed analysis (BOD<sub>30</sub>) cannot be run any faster and samples requiring immediate analysis (bacteria) will be done immediately anyway.

#### **DIRECTIONS TO DCLS**

DCLS is located at 600 North 5<sup>th</sup> Street Richmond, VA 23219

Temporary parking is available for sample delivery at the DCLS loading dock/sample receiving at 600 North 4<sup>th</sup> Street. Ring buzzer by door to right of loading dock doors for entrance into the loading dock area. From West of Richmond:

- 1: Start out going East on I-64 E.
- 2: Take the I-64 E exit- Exit 75- toward WILLIAMSBURG/NORFOLK. 0.17 miles
- **3:** Take the 3RD STREET ramp toward COLISEUM/DOWNTOWN. 0.09 miles
- 4: Stay straight to go onto N 3RD ST. 0.13 miles
- **5:** Turn LEFT onto E LEIGH ST. 0.06 miles
- 6: Turn LEFT onto N 4TH ST. 0.04 miles. Sample receiving is in the middle of the block on the right.

#### From South of Richmond;

- 1: Start out going North on I-95 N.
- 2: Take the CHAMBERLAYNE AVE exit- Exit 76A. 0.16 miles
- 3: Turn LEFT onto CHAMBERLAYNE AVE/CHAMBERLAYNE PKWY. 0.20 miles
- **4:** Turn SLIGHT LEFT onto W LEIGH ST. 0.30 miles
- 5: Turn LEFT onto N 4TH ST. 0.04 miles. Sample receiving is in the middle of the block on the right.

#### From East of Richmond;

- 1: Start out going West on I-64 W toward RICHMOND.
- 2: Take the I-95 S/5TH STREET exit- Exit 190- on the left toward

PETERSBURG/DOWNTOWN/COLISEUM, 0.29 miles

- 3: Stay straight to go onto N 5TH ST0.12 miles
- **4:** Turn RIGHT onto E JACKSON ST0.12 miles.
- 5: Turn LEFT onto N 3RD ST. 0.07 miles
- **6:** Turn LEFT onto E LEIGH ST. 0.06 miles.
- 7: Turn LEFT onto N 4TH ST. 0.04 miles. Sample receiving is in the middle of the block on the right.

#### From North of Richmond;

- 1: Start out going South on I-95 S toward RICHMOND.
- 2: Take the I-64 E exit- Exit 75- toward WILLIAMSBURG/NORFOLK. 0.17 miles
- 3: Take the 3RD STREET ramp toward COLISEUM/DOWNTOWN. 0.09 miles
- **4:** Stay straight to go onto N 3RD ST. 0.13 miles
- **5:** Turn LEFT onto E LEIGH ST. 0.06 miles
- **6:** Turn LEFT onto N 4TH ST. 0.04 miles. Sample receiving is in the middle of the block on the right.

#### **APPENDIX E**

## **BACKUP SAMPLE DROP-OFF PROCEDURES**

Revised 04-01-2004

Note: In December 1998 DCLS contracted a courier to pick up samples from all the regions. The following sample drop-off procedures will be used only in cases where DCLS courier is unavailable.

#### I. Schedule

Tributary sampling occurs once each month for each river:

#### II. General Procedures

As far as possible in advance, contact the appropriate personnel at each region affected to coordinate the time of day you will meet and pick up the samples. Drop off samples at DCLS after 8:00 am via the warehouse. Sign the samples in at the desk.

#### York River:

Nothing. TRO will either deliver samples to PRO who will take the samples to DCLS or TRO will leave the sample coolers at boat dealership adjacent to public ramp of Coleman Bridge and PRO will transport to DCLS.

#### Non-general procedures for York River:

If PRO and TRO do not go out on the same day, a pick-up has to be made for the TRO samples at West Point. A car needs to be reserved and after you drive through West Point on Rt.33, just before the Mattaponi on the left hand side is a road going to the boat ramp. Either meet TRO there or they will drop off the samples (there is a fenced in boat building near the boat ramp and the samples are left just inside the fence). Samples are taken to DCLS.

#### James River:

**TRO:** Meet TRO at Jamestown at 11:00 (drive time 1 hr, 15 min.). Directions to Jamestown are:

Take 64 East to Exit 242A (Rt.199 West, Williamsburg, Busch Gardens). Take left on 31 south, left on 359 (Jamestown), left into parking lot, through lot turn right into marina. TRO samples will need to be transferred into the cooler and TRO will supply a cooler for the chlorophyll samples. Return to Richmond, take samples to DCLS

#### Rappahannock River:

TRO: Note: In 2010, PRO began sampling the stations formerly sampled by TRO on the Rappahannock. TRO no longer samples the Rappahannock; the following directions are for documentation purposes.

Meet TRO at Locklies marina at 10:00 - 10:30 (drive time 1 - 1 1/2 hours). Directions - Take 64 east to 33 East (West Point). Take 33 to Rt.17 North. Take 17 North about 2 miles to 17 Business into Saluda. Continue on 17 Business into town, turn right at red light (Rt.33) Take 33 to Rt.3 West (turn left). Turn right onto Rt.621 (just after small airport. Locklies is at end of 621.

NRO: Note: In 2003 NRO changed their launch/retrieval ramp to one behind the Little Falls Sewage Treatment Facility on Route 3. The following directions are for the Fredericksburg city dock and are provided for documentation purposes only. Meet NRO at Fredericksburg at 12:30 -1:00 (drive time from Locklies 1 1/2 hours). Directions - retrace back 621, 3, and onto 33. When into Saluda, go straight through red light. A mile further down the road will be Rt.17. Turn right (north) on 17. Go through Tappahannock (PRO launches at Hoskins Creek - Dock St., you may want to contact them at this time). Go into Fredericksburg (Rt 2 & 17 Business), Turn left on Rt.17 Truck. Go under Railroad bridge and turn right at light (Lafayette). Go to end of road (two blocks) and turn right (Sophia). Go under the railroad bridge and you will be at the public boat launch area In 2018 NRO was forced to again relocate the launch site due to sedimentation build up on ramps. A private ramp ~ 12 miles from I95 is currently the launch/retrieval site. Take Rt. 3 east for 11 miles and turn onto Brenthem Farm Drive.

**PRO:** Note: The following directions are for documentation purposes, and are no longer current.

For PRO you can do one of three things.

- 1) Let them deliver the samples to DCLS.
- 2) Meet PRO at the Hoskins Creek boat landing in Tappahannock. Directions: Travel south from Fredericksburg on Rt.17 to Tappahannock. Just before Hoskins Creek is a 7-11 store/gas station on the left, turn there, PRO should be finished around 3:00).
- 3) Meet PRO at Texaco station.

Directions: Go south on 95 (you can get gas at Atlee if you need gas). Take 295 East (towards Norfolk), then take 360 West. There is a Texaco station just over the overpass on the left. Wait for PRO there. Take samples to DCLS.

#### Rappahannock River: (If only one region is sampling)

Note: In 2010, PRO began sampling the stations formerly sampled by TRO on the Rappahannock. TRO no longer samples the Rappahannock; the following directions are for documentation purposes.

**TRO:** Meet at Locklies, take one extra cooler (for DCLS samples), deliver samples to DCLS.

**PRO:** Meet at Texaco station at Rt.295 and Rt.360 (they can call when leaving Hoskins Creek, it will take them a little longer to get to the Texaco station then for you to get there from downtown). Pick up DCLS samples or have PRO take samples to DCLS.

# APPENDIX F CBP Required Parameter list

Revised 06-11-07

## Field Parameters:

## **Storet Code Description**

00010	Temperature in Celsius Deg.
00078	Secchi in Meters
00094	Specific Conductance (umhos/cm at 25 C)
00299	Dissolved Oxygen via probe (mg/L)
00096	Salinity
EPARS *plankton sites	Photosynthetically Active Radiation obtained at surface with Li-cor Unit
only	
EPARD*plankton sites only	Photosynthetically Active Radiation obtained below surface with Li-cor
	Unit

## Analytical requirements:

32211	Chlorophyll a, corrected, monochromatic	
32218	Pheophytin, corrected, monochromatic	
630BX	Optical Density obtained Before addition of HCl	
647BX	Optical Density obtained Before addition of HCl	
664BX	Optical Density value obtained Before addition of HCl	
665AX	Optical Density value obtained After addition of HCl	
71994	Volume filtered in L	
750AX	Optical Density value obtained After addition of HCl	
750BX	Optical Density obtained Before addition of HCl	
CELLP	Cell path length in cm	
EXTVO	extraction volume in mL	
00530	Total Suspended Solids	
00535	Volatile Suspended Solids – not submitted to CIMS	
00540	Fixed Suspended Solids	
00608	Dissolved Ammonia (mg/L as N)	
00613	Dissolved Nitrite (mg/L as N)	
00618	Dissolved Nitrate (mg/L as N) – not submitted to CIMS	
00631	Dissolved Nitrite plus Nitrate (mg/L as N)	
00671	Dissolved Orthophosphorus (mg/L as P)	
00955* plankton sites only	Dissolved Silica (mg/L as SiO2) – value divided by 2.14	
	prior to submittal to CIMS	
49571	Total Dissolved Nitrogen (mg/L)	
49572	Total Dissolved Phosphorus (mg/L)	
49569	Particulate Carbon (mg/L)	
49570	Particulate Nitrogen (mg/L)	
49567	Particulate Phosphorus (mg/L)	
49573* plankton sites only	Dissolved Organic Carbon	