STANDARD OPERATING PROCEDURE FOR ELIZABETH RIVER PROGRAM CRUISE DEPLOYMENT

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DISCLAIMER: This SOP applies to cruise deployment for field monitoring and sample collection in support of the USEPA Chesapeake Bay Water Quality Monitoring Program for samples collected in the Elizabeth River. This SOP may not be applicable to any other studies.

TABLE OF CONTENTS

1. LOCATION

This procedure will be used by Water Quality Laboratory staff during cruise deployment on the Elizabeth River.

2. PURPOSE

This procedure is designed to ensure that all protocols are followed during physical profile measurements, sample collection and onboard processing of samples during field operations. The intended data user groups are the subcommittees and workgroups of the USEPA Chesapeake Bay Monitoring Program (ER). These data will be used to assess whether the multijurisdictional action plan for restoring the water quality of the Chesapeake Bay is effective. This assessment is performed, in part, by analysis of the data, and its inclusion in computer models of the Chesapeake Bay. Trend analyses are also performed on the data to assess whether Chesapeake Bay restoration goals are being met. These data may also be used to by State and Federal government officials to pass legislation designed to help improve the quality of the water in the Chesapeake Bay and its tributaries.

3. APPLICABILITY

This procedure is applicable to field operations conducted on the Elizabeth River by the WQL.

4. OVERVIEW

This SOP is based on the procedures developed by Old Dominion University in support of field operations.
The physical profile of the station is measured using a YSI 6000UPG which is calibrated for temperature, salinity, conductivity, pH, depth and dissolved oxygen. The YSI is lowered into the water column attached to an array containing a photometric sensor that measures ambient light and a submersible pump which is fitted with a polyethylene hose which is used to collect ambient water samples. The water samples are processed as soon as possible to ensure that they are properly preserved for movement to the laboratory.

5. DEFINITIONS AND ABBREVIATIONS

NO_3:	Nitrate
NO_2:	Nitrite
L:	Liter(s)
mL:	Milliliter(s)
g:	Gram(s)
mg:	Milligram(s)

mg/L:	Milligram(s) per liter
N:	Normality of the chemical solution (g/L)
M:	Molarity of the chemical solution (mol/L)
CBP:	Chesapeake Bay Monitoring Program
ER:	Elizabeth River Monitoring Program
USEPA:	United States Environmental Protection Agency
HDPE:	High-density polyethylene

6. CHEMICALS USED

Chemicals Used: All chemicals must be analytical grade or of a higher purity except as noted.

6.1 For Winkler Dissolved Oxygen standardization and titration.
Manganous sulfate ($MnSO_4 \cdot H_2O$)
Potassium iodate granular (KIO_3)
Potassium iodide (KI)
Salicylic acid (2-Hydroxybenxoic acid)
Sodium azide (NaN_3)
Sodium hydroxide (NaOH)
Sodium iodide (NaI)
Soluble starch ($C_6H_{10}O_5$)
Sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$)
Sulfuric acid (H_2SO_4)

6.2 For Chlorophyll preservation.
Magnesium carbonate ($MgCO_3$)

Safety Equipment for on-board procedures:

1. **Protective clothing should be worn when handling strong acids and bases, i.e. SULFURIC ACID, SODIUM HYDROXIDE**
2. **Protective eye wear: Goggles when handling liquids, glasses are approved for handling solids only**
3. **Nitrile gloves**

Procedure will be conducted in a well ventilated area

Note: Potassium iodide, sodium azide, and sodium iodide emit toxic vapors when heated. Sodium azide is a very toxic poison by dermal exposure as well as other routes. **All skin contact with sodium azide should be avoided.**

NOTE: Refer to Safety Precautions and use care when preparing reagents.

6.3 Before and after preparing reagents, wipe down the counter with ultrapure water and a paper towel.

6.4 Clean balance and balance table before and after each use using ultrapure water and a paper towel, and/or brush.

6.5 On each reagent bottle the following information must be recorded:

Reagent Identification (name) and Concentration:
Analysis: (Which analysis requires this reagent?)
Date prepared:
Prepared by:
Expiration date: (put N/A if there is no holding time)
Storage requirements: (i.e. room temperature, 4° C)

6.6 Preparation of Reagents needed for the analysis:

Inspect the reagents required for sufficient quantity, expiration dates and appearance.

If necessary remake reagents following the procedures in the SOP entitled Winkler Dissolved Oxygen and Chlorophyll: Spectrophotometric Method.

NOTE: Refer to Safety Precautions and use care when preparing reagents.

6.7 Reagent containers are used only for the intended reagent and are reused. Before fresh reagents are added to the containers, the containers are rinsed 9 times with ultrapure water, and 3 times with the fresh reagent.

7. ON BOARD PREPARATION

The following is a guide for setting up equipment onboard the research vessel. These specific instructions are for the R/V Miss Jana. If another research vessel is utilized, the positions of the equipment on the vessel may be shifted according to need and maximizing efficiency. The onboard preparation is performed before or on the morning of the cruise.

7.1 Sample Carboys - Two sample carboys are located outside with the field technician operating the array. These carboys are used in sample collection for the individual parameters and are labeled S and B.

7.2 Submersible Pump - The barrel containing the submersible pump and polyethylene hose is positioned next to the winch toward the center portion of the boat on the port side. The power cord for the pump, the light meter and YSI cables are attached to the hose with duct tape.

7.3 Set up the field array in the vicinity of the winch and hose bucket. See the light attenuation and YSI SOPs for details.

7.4 Cruise boxes for the pumps should be stored underneath the laboratory table area. The pumps are placed on the counters in the laboratory.

7.5 The filtration boxes containing filtration flasks are placed in front of the vacuum pumps on the laboratory table. Make sure the outlet hose from the filtration box is attached to the top of the waste flask and that the waste water level never exceeds either the side arm or the piece of pipette in the stopper.

7.6 The miscellaneous box is stored in the laboratory for easy access by field technicians during the course of the cruise.

7.7 The Winkler dissolved oxygen reagents for the initial sample preservation are contained in a tackle box . The plexiglas filter holder box containing concentrated sulfuric acid, AIA, $MnCl_2$ and starch is placed on the counter. The digital buret containing sodium thiosulfate

is secured next to the TSS/PPO_4 filtration box in a wooden box. The stirplate is placed in front of the digital buret. A 1 mL Eppendorf pipet, approximately 20 pipet tips and several plastic transfer pipets are located in a bag and placed in the sink, near the 100 mL plastic graduated cylinder, stirbar and stirbar retriever. As conditions indicate, this equipment may be secured with duct tape to the counter.

7.8 One of the carboys containing Ultrapure water is placed underneath the laboratory table. The other is placed forward in the hold.

7.9 The box containing the filters, sample vials and filtering apparatus for processing PC/PN samples is placed underneath the laboratory table.

7.10 All of the sample bottles are stored in bags in the laboratory.

7.11 The extra YSI is stored forward on the boat.

8. RESEARCH VESSEL DUTIES THE DAY OF THE CRUISE

8.1 The Chief Scientist reports the names of the crew to the Captain of the research vessel. Weather is discussed and a decision is made as to whether to leave the dock.

8.2 The research vessel departs.

8.3 Onboard preparation is performed, if not completed earlier.

8.4 The data sheets and cruise log are started for the day. See Appendix 1.

8.5 Captain informs Chief Scientist that the boat is on station and sampling activities begin. Members of the crew perform assigned duties simultaneously.

9. ON-STATION ACTIVITIES

9.1 Determine the Secchi Depth immediately upon reaching station:

9.1.1 The 20 cm secchi disk is attached to a brass chain which is marked in 0.1 m increments with colored plastic marks.

9.1.2 Lower the secchi disk into the water on the shady side of the boat. If overcast, then at discretion of technician.

9.1.3 Continue lowering the secchi disk until the black and white quadrants are no longer distinguishable. Note this depth.

9.1.4 Slowly retrieve the secchi disk, noting the depth in which the black and white quadrants are once again distinguishable.

9.1.5 The secchi depth is the average of the depths observed in Steps (9.1.3) and (9.1.4). This depth is recorded onto the field data sheet to the closest 0.1 m.

9.2 The Chief Scientist enters station identification and time of day in military time on the CBP monitoring form and the field data sheet. See Appendix 1 for an example of a completed field data sheet.

9.3 The weather is recorded on the data sheet.
The weather codes are as follows:

Cloud Cover	Precip. Type	Wind Speed	Sea State
0-Clear (0-10%)	10-None	0 calm	0 calm
1-Partially cloudy(10-50%)	11-Drizzle	1 1-10 kt	1 $\leq$ 1 ft
2- Partially cloudy,(50-90%)	12-Rain	2 10-20 kt	2 $\leq$ 2 ft
3-Overcast (> 90%)	13-Rain Heavy	3 20-30 kt	3 $\leq$ 3 ft
4-Foggy	14-Squally	4 30-40 kt	4 $\leq$ 4 ft
5-Hazy	15-Frozen Precip	5 > 40 kt	5> 4 ft
6-Cloud (no percentage)	16-Rain Snow		

*The wind direction is also recorded on data sheet, but there is no code. (Recorded as : N, NE, SSW, etc)

9.4 The current location of the research vessel is determined using the GPS from the Research Vessel.

9.5 The physicochemical profile is measured using the YSI and recorded onto field data sheets. The physicochemical profile is performed by one person lowering the probe(s) to the proper depth (using YSI depth sensor) while one person takes the instrument readings according to theprocedures outlined in the applicable SOPs. See Appendix 1 for an example of a completed field data sheet.

CAUTION: DISSOLVED OXYGEN MEASUREMENTS MUST BE TAKEN FROM SURFACE TO BOTTOM BY LOWERING THE PROBE THROUGH

THE WATER COLUMN.

9.6 Water samples are collected using the submersible pump attached to the sampling array:

9.6.1 The surface sample is pumped from 1 m below the surface into the sample carboy labeled S.

9.6.2 Once the pump is positioned at the proper depth, the hose is flushed for 1 minute or until the bubbles have completely cleared the line.

9.6.3 The sample carboy is allowed to completely fill and then emptied twice to rinse. Open stopcock briefly to flush with sample.

9.6.4 The hose is placed in the top of the sample carboy allowing it to fill completely.

9.7 Record physiochemical profile following the instructions in the YSI SOP.

9.8 Record light attenuation data following instructions in the light attenuation SOP.

9.9 Repeat Steps (9.7) through (9.8), lowering the array in 0.5 m intervals until Channel 2 and Channel 3 for light attenuation are < 1. Then, lower the array in 1 m intervals up to 15 m and then at 2 m intervals thereafter for each measurement. **Note: YSI data is recorded at every meter up to 15 m and then at every 2 meters. Light attenuation data is recorded at every 0.5 m until Channel 2 are < 1.**

9.10 Only for plankton stations: The presence or absence of a pycnocline is determined at designated stations using the equations selected by the EPA CBP as follows:

$$\frac{\text{surface cond.}}{\text{bottom cond.}} = \text{cond.}$$

$$\frac{(\ \text{cond})}{(\text{depth m}) - 0.05} = \text{x } 2 = \text{(pycnocline threshold)}$$

Upper limit = shallowest depth where change in cond. $\geq$ pycnocline threshold
Lower limit = deepest depth where change in cond. $\geq$ pycnocline threshold

Note: No pycnocline if 1) pycnocline threshold < 0.5 millihos/cm, or 2) no depth interval is > threshold value.

9.11 The last measurements are taken 1 m above the bottom. When all measurements are completed, the hose is disconnected from the manifold and water from the thru- hull is allowed to flow through the fluorometer.

9.12 The bottom sample is collected 1 m above the bottom in the same manner as the top sample carboy.

9.13 The Chief Scientist completes the CBP monitoring form and the chief scientist check list (appendix 1).

9.14 When all necessary station activities are complete, the Chief Scientist directs the Captain of the research vessel to move to the next station.

9.15 This procedure continues until all stations have been sampled.

10. ACTIVITIES FOLLOWING SAMPLE COLLECTION

These procedures should be performed as soon as possible after collection.

10.1 **WINKLER DISSOLVED OXYGEN:** At designated stations assigned by the Chief Scientist, an aliquot of a water sample is collected in a 300 mL BOD bottles for Winkler DO titration using the following procedures. A minimum of two winker dissolved oxygen samples should be collected per sampling day. This is to ensure the YSI dissolved oxygen values are accurate. If the winkler dissolved oxygen value and the YSI dissolved oxygen value do not compare, steps need to be taken to identify the problem. See section 10.14 for guidance on comparing dissolved oxygen values and steps to rectify any discrepancy.

NOTE: It is essential to collect samples and fill the sample bottles in a manner that will avoid aeration and/or sample turbulence.

10.1.1 Ensure that the Tygon® tubing on the stopcock of the sample carboy is clean and is long enough to reach to the bottom of the BOD bottle.

10.1.2 Rinse the BOD sample bottle 3 times discarding the rinsate.

10.1.3 Invert the BOD bottle with the subsampling hose touching the edge where the side and bottom of the bottle join.

10.1.4 Open the stopcock on the sample carboy and adjust for a slow flow rate. Orient the

sample bottle to achieve complete rinsing of the entire inside walls of the bottle. Rinse for approximately 15 seconds.

10.1.5 After rinsing, **SLOWLY** turn the BOD bottle to an upright position for filling, keeping the filling hose submerged at all times. If any bubbles are observed during filling, start over using a slower fill rate.

10.1.6 After sample bottle is full, keep filling to allow the sample to overflow (approximately 25-50 mL).

10.1.7 Immediately perform steps 10.1.8 through 10.1.10.

10.1.8 Immediately add 1 mL of $MnSO_4$ solution, followed by 1 mL of alkali-iodide-azide (AIA) reagent.

10.1.9 Place the glass stopper onto the sample bottle to avoid introducing air bubbles. Fit plastic cap with foam insert onto glass stopper to prevent spilling.

10.1.10 Invert the sample bottle 3 times to mix. This must be done immediately after sample is collected.

10.1.11 When the precipitate has settled to at least half the sample bottle volume (leaving clear supernatant above the manganese hydroxide floc), add 1mL of H_2SO_4 and mix the sample again by inverting the bottle 3 times.

10.1.12 Follow the instructions for titration method in the Winkler DO SOP Titration must be completed within 8 hours of sample collection.

10.2 **TSS/PPO_4:** An aliquot of each water sample is collected in a 2L labeled sample bottle for concentration of total suspended solids (TSS), particulate phosphate (PPO_4) and filtration of nutrient water samples using the following procedures.

10.2.1 Using the sample carboy, rinse the entire inside surface of the 2000 mL sample bottle three times with sample, discarding the rinsate.

10.2.3 Fill the sample bottle with sample, leaving approximately one inch of air space.

10.3 **CHLOROPHYLL:** An aliquot of each water sample is collected in a 1000mL brown bottle for chlorophyll concentration using the following procedures.

10.3.1 Rinse the entire inside surface of the sample bottle three times with sample, discarding the sample before successive rinses.

10.3.2 Fill the sample bottle with sample, leaving approximately one inch of air space in the sample bottle.

10.3.3 Immediately mix the magnesium carbonate suspension and add 1 mL per liter of sample. Invert the sample to mix (see definitions). Protect sample from direct sunlight. If the samples can not be immediately filtered, store at 4ºC. Samples must be filtered within 24 hours.

10.4 **PC/PN:** An aliquot of each water sample is collected into a labeled 250 mL glass bottle for PC/PN analysis.

10.4.1 Rinse the entire inside surface of the sample bottle three times with sample, discarding the rinsate.

10.4.2 Fill the sample bottle with sample, leaving approximately one inch of air space in the sample bottle.

10.5. The samples should be filtered immediately after collection. If this is not possible, store samples at 4° C or lower 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 cap. Storage temperature must be measured using a thermometer which has been calibrated against an NIST-traceable thermometer within the last six months. Storage temperature is documented on the CBP monitoring form.

10.6 **TSS/PPO_4:** The water sample collected in the 2L labeled sample bottle for concentration of total suspended solids (TSS) and particulate phosphate (PPO_4) analysis and filtration of nutrient water samples is processed using the following procedures:

10.6.1 Labware needed:

- 4- filtration towers
- 4- 2000 mL or 4000 mL filtration flasks
- 2- forceps

- 4- 500 mL graduated cylinders
- 2- filter holders containing pre-rinsed 4.7 cm Whatman®GF/F glass fiber filter (at least one of which is pre-weighed for TSS) for each sample

1- filter holder for every 10 samples containing a pre-rinsed 4.7 cm Whatman® GF/F glass fiber filter for a PP duplicate
1- filter holder for every 10 samples containing a pre-rinsed, pre-weighed 4.7 cm Whatman®GF/F glass fiber filter for a TSS duplicate

NOTE: The procedure for preparing filters which will also be used for total suspended solid analysis is in the SOP entitled "**Standard Operating Procedure for Total Suspended Solids Dried at 103-105°C**".

NOTE: For the procedure for preparing filters which will also be used for particulate phosphate analysis is in the SOP entitled "**Standard Operating Procedure for Particulate Phosphate in Water and Seawater Using A SKALAR Autoanalyzer**".

10.6.2 Using forceps, gripping only the filter edge, transfer a pre-rinsed and pre-weighed (if also analyzing for total suspended solids) 4.7 cm Whatman®GF/F glass fiber filter (or equivalent) with the wrinkled side up onto the base of a filtration tower. Replace the top of the filtration tower onto the base. Make sure the filtration tower is on the back row of the TSS/PPO_4 manifold.

10.6.3 Moisten the filter with fresh ultrapure water. Move the filtration tower from the back row to the front to collect the filtrate in a 2L filtration flask.

10.6.4 Rinse the entire inside surface of a 500 mL graduated cylinder three times with ultrapure water. Mix the sample thoroughly by inverting (see definitions) the sample bottle several times until well-mixed. Immediately rinse the graduated cylinder twice with the sample to be filtered, discarding the rinsate.

10.6.5 Immediately pour a pre-determined volume of sample into the rinsed graduated cylinder. **Record the filtration volume in the appropriate space on the total suspended solids data sheet.**

10.6.6 Filter the sample water through the filter using 20 KPa vacuum pressure. To avoid cell damage during filtration, do not exceed this vacuum pressure, and limit filtration duration to 10 minutes or less. If it takes longer than 10 minutes to filter, discard filter and remaining sample, rinse following step 10.6.15, then complete steps 10.6.2 through 10.6.12 using a lesser sample volume. **Record the new volume on the total suspended solids/particulate phosphate data sheet and initial the change.**

10.6.7 When sample filtration is complete, move the filter and filtration tower to the back row of the manifold.

10.6.8 Apply vacuum pressure of 20 KPa. Completely rinse all inside surfaces of the graduated cylinder with fresh ultrapure water. Pour the ultrapure water into the filtration tower.

10.6.9 Rinse the graduated cylinder again, following step 10.6.4.

10.6.10 Rinse filter with approximately 20 mL of fresh ultrapure water.

10.6.11 Rinse the filter 2 more times, following step 10.6.4, allowing each aliquot of water to pass through the filter before successive rinses.

10.6.12 Using forceps, being careful not to touch or disturb the filtrate, remove the filter from the filtration tower, fold in half, and place it in a filter holder which is labelled with the Project ID, sample ID and sample log number. Place the cover on the filter holder.

10.6.13 Store the holder in a water-tight container packed in ice and freeze as soon as possible.

10.6.14 See temperature documentation requirements in step 10.5.

10.6.15 Completely rinse the empty filtration tower three times with fresh ultrapure water before filtering the next sample.

10.6.16 Repeat steps 10.6.2 through 10.6.13 for each sample, using an unweighed filter that has been prepared for particulate phosphate analysis, recording the volume filtered on the total suspended solids/particulate phosphate filtration logsheet under the column heading " **PP Vol**".

NOTE: Filter one extra randomly chosen sample for every 10 samples, following steps 10.6.2 through 10.6.13 and record the volume filtered on the particulate phosphate filtration logsheet under the column heading " PP DUP Vol".

10.6.17 The water collected in the filtration flask is divided into 3 sample bottles as follows:

1- 250mL bottle with split code -01 (for NO3, NH3)
1- 250mL bottle with split code -02 (for OPO4, NO2)
1- 125mL bottle with split code -08 (for TDN, TDP)

Rinse the entire inside surface of each sample bottle labeled with the sample's log number and sample ID three times with the filtered sample.

Pour the filtered water into the sample bottle, leaving approximately one inch of air space in the bottle.

Pack the sample bottles in ice in coolers up to the level of the top of the sample but below the bottom of the sample bottle cap. See temperature documentation requirements in step (10.5).

Discard the remaining filtrate after subsamples for all parameters have been collected. Completely rinse the empty filtration flask and tower three times with fresh ultrapure water before filtering the next sample.

Repeat steps 10.6.17 for each sample.

10.7 **CHLOROPHYLL:** The water sample collected in a 1000mL brown bottle for chlorophyll concentration is processed using the following procedures.

10.7.1 Using forceps, gripping only the filter edge, transfer a 4.25 cm Whatman®GF/F glass fiber filter (or equivalent) with the wrinkled side facing up, onto the base of a filtration tower. Replace the top of the filtration tower onto the base.

10.7.2 Rinse the entire inside surface of a 500 mL graduated cylinder three times with fresh ultrapure water. Mix the sample thoroughly by inverting (see definitions) the sample bottle several times until well-mixed. Immediately rinse the graduated cylinder twice with the sample to be concentrated.

10.7.3 Immediately pour a pre-determined volume of sample into the rinsed graduated cylinder. **Record the sample volume that was filtered in the appropriate space on the benchsheet.**

10.7.6 Concentrate the plankton by filtering the sample water through the filter using 10 psi vacuum pressure. To avoid cell damage during filtration, do not exceed this vacuum pressure and limit filtration duration to 5 minutes or less. If it takes longer than 5 minutes to filter the selected sample volume, discard filter and remaining sample, rinse the filtration apparatus (see step 10.2.9), then complete steps 10.2.2 through 10.2.6 using a lesser sample volume. **Record the new volume on the chlorophyll data sheet and initial the change.**

10.7.5 Fold the filter in half using forceps, being careful not to touch or disturb the filtrate. Fold the filter a second time lengthwise and place the folded filter on a labeled square of aluminum foil and seal tightly.

10.7.6 Store in a sealed plastic bag and place in the freezer in the red refrigerator. See temperature documentation requirements in step 10.5.

10.7.7 Completely rinse the empty filtration tower three times with fresh ultrapure water before seating a new filter for the next sample.

10.7.8 Repeat steps 10.7.1 through 10.7.7 for each sample.

NOTE: Filter one randomly chosen sample in duplicate for every 10 samples, and filter one extra sample duplicate for every sampling event, following steps 10.3.1 through 10.3.8.

10.8 **PC/PN:** The water sample collected for concentration of particulate carbon/particulate nitrogen (PC/PN) in a 250 mL glass bottle is processed using the following procedures.

10.8.1 PC/PN filter holders should be loaded in the laboratory, but can be loaded as needed on the boat. Using forceps, gripping only the edge, transfer a muffled 13mm Whatman GF/F glass fiber to the top of the filter holder making sure the O-ring is in place. Screw the bottom of the holder on to the top of the holder.

10.8.2 Rinse the entire inside of the 50 mL graduated cylinder three times with ultrapure water. Mix the sample thoroughly by inverting (see definitions) the sample bottle several times until well-mixed. Immediately rinse the graduated cylinder twice with the sample to be concentrated.

10.8.3 Immediately pour the pre-determined volume of sample into the rinsed graduated cylinder.

10.8.5 Record the filtration volume, sample ID and Log number in the appropriate spaces on the bench sheet.

10.8.6 The volume filtered will depend on the amount of suspended solids present in the sample, usually 30-50 mL.

10.8.7 Pour sample from the graduated cylinder into a 60cc syringe with an attached filter tip containing a muffled 13mm glass fiber filter.

10.8.8 Place the syringe on the top of the filtration tower attached to the PC/PN - Chlorophyll manifold . Attach manifold to a vacuum pump set to $\leq$ 15 psi vacuum.

10.8.9 Repeat so that each sample is filtered in duplicate. Filter 6 triplicate samples per cruise day; 18 triplicates total.

10.8.10 Remove filter using clean metal curved forceps from the filter holder without touching the area containing sample, fold in quarters, and place each filter into individually labeled, muffled glass vials with teflon-lined screw caps.

10.8.11 Place vials in divided cardboard box and place on ice.

10.9 The Chief Scientist ensures that all samples are properly collected and checks all recorded data and calculations for completeness, legibility, and verification that all data appears to be valid. If there are any questions, measurements in question will be repeated for verification.

10.10 All samples, except Winkler D.O. samples, are then stored in an ice chest at or below 4°C. Ice must be in contact with all surfaces of each sample container below the neck.

10.11 When the Chief Scientist has confirmed that all samples have been properly collected, processed, stored, all required data has been recorded on field data sheets and the chief scientist check list (appendix 1), the samples in the sample carboys can be discarded and preparation for the next sampling event begins. **Note that only the chief scientist can authorize discarding samples or leaving station.**

10.12 As time allows between stations:

10.13.1 Winkler DO samples are titrated. See the Winkler DO SOP for proper procedures.

All information and data is recorded on the data sheet.

10.13.2 The BOD bottle for Winkler DO titration are titrated in the following manner.

10.13.3 Allow the floc to resettle to at least half the sample bottle volume. However, steps 10.3.4 through 10.13.10 must be completed within 8 hours of sample

collection.

10.13.4 Stir the sample using a stirbar and magnetic stirplate.

10.13.5 Add 1 mL of concentrated sulfuric acid (H_2SO_4) and stir slowly and carefully.

10.13.6 Slowly and carefully after dissolution is complete, pour off 99 mL of the solution using a 100 mL graduated cylinder. Sample will have deep yellow-orange color. It is critical to avoid sample turbulence and aeration during this step or else iodine could be lost from the sample due to its extreme volatility, resulting in erroneously low analytical results.

10.13.7 Put the sample back on the stirplate and, while stirring, titrate the sample with 0.210 M $Na_2S_2O_3$ solution to a pale straw color (using a titrator or buret capable of 0.01 mL resolution). **In order to achieve maximum accuracy, the color should be as pale as possible without becoming colorless.**

10.13.8 Add a few drops of starch solution and continue titration to the first disappearance of blue color.

10.13.9 If the samples does not turn blue after adding starch indicator the endpoint was exceeded in step 10.13.7. Add volumetrically 0.210M potassium iodide (KIO_3) solution, using an Eppendorf®pipette, without delay to re-color the sample (correction for this volume as indicated in the equation below).Record the amount of KIO_3 in the appropriate space on the data sheet.

10.13.10 Complete the titration to a clear endpoint without delay.

10.13.11 After completing the titration, disregard subsequent re-coloration. This is due to the catalytic effect of nitrite or traces of ferric salts that have not been complexed.

10.13.12 Winkler D.O. reagents should be checked monthly and should be replaced when either color changes, foreign objects are observed, or holding times are exceeded.

10.14 Chief Scientist will compare the DO value of the winkler DO to the YSI values. If the difference is greater than 0.5 mg/L the Chief Scientist will do the following;

10.14.1 Increase supervision of all steps.

10.14.2 Verify that samples are consistently being collected properly and provide additional training as necessary.

10.14.3 Verify that the probe membrane does not have air bubbles beneath it or otherwise does not need to be changed.

10.14.4 Verify that the reagents do not have an atypical appearance and are within their shelf life.

10.14.5 Verify that reagents are consistently being added to samples properly and provide additional training as required.

10.14.6 Verify that samples are being titrated to the appropriate endpoint. Provide additional training as necessary.

10.14.7 Include description of problem and all observations in field log and cruise report.

10.14.8 If cannot identify any obvious problem with winkler dissolved oxygen procedure, switch to the back up YSI sonde to see if this rectifies the problem. This instrument switch should be noted on the CBP MONITORING FIELD SUMMARY form.

10.14.9 Inform the Lab Supervisor promptly after the cruise.

10.15 At an arbitrary point determined during each cruise one field blank is processed near the end of the ER cruise. One liter of ultrapure water is filtered following the same procedures and protocols used for samples. The filter is analyzed for total suspended solids and PPO_4.

10.17 The filtered water is collected and poured into the same bottlesas the nutrient samples and stored with cruise samples. In order for the necessary information to be properly and validly obtained, no special treatment or handling such as extra rinsing of the filtration apparatus, may occur. The filtered Ultrapure water is collected for NO_3+ NO_2, NH_3, DPO_4, and TDN.

10.18 At the same time the field blank is filtered for nutrients, 50 mL of Ultrapure water is filtered through a muffled Whatman® GF/F 13 mm filter and the filters are analyzed for PC/PN.

11. Appendix 1 - Example of a Completed Field Data Sheet

CHESAPEAKE BAY MONITORING PROGRAM

STATION: SBE5	COMMENTS:	RV: MISS JANA	
	CRUISE NO.: ER115	DATE: 8/12/98	TIME: 0927
FIELD CHIEF: S.LEE	STATION DEPTH: 6 M	LAT. 36 45.885	N
	SECCHI DEPTH: 1.0 M	LONG. 76 17.975	W

WEATHER DATA - CIRCLE APPROPRIATE WEATHER CODES

CLOUD COVER	PRECIP. TYPE:	WIND SPEED	SEA STATE
0 - CLEAR (0-10%)	10 - NONE	0 0 - 1 KNOTS	0 CALM
1 - PARTIALLY CLOUDY: 10-50%	11 - DRIZZLE	1 2 - 10 KNOTS	1 < 1 FT.
2 - PARTIALLY CLOUDY: 50-90%	12 - RAIN	2 11 - 20 KNOTS	2 < 2 FT.
3 - OVERCAST: >90%	13 - RAIN HEAVY	3 21 - 30 KNOTS	3 < 3 FT.
4 - FOGGY	14 - SQUALLY	4 31 - 40 KNOTS	4 < 4 FT.
5 - HAZY	15 - FROZEN PRECIP.	5 >40KNOTS	5 > 4 FT.
6 - CLOUD (NO PERCENTAGE)	16 - RAIN SNOW		
		WIND DIRECTION	TIDAL STAGE: H L F E
		NONE	

ZOOPLANKTON NET DATA

DROP NET VOLUME (ML) A: B

NET:	METER STOP	METER START	TOTAL REVS.	
A:	574541	570000	4541	TOW TIME: 4 MIN 30 SEC
B:	493996	490000	3996	

MESOGLEA DATA

MESOGLEA VOL.	A	B		A	B
(TOTAL)	200 ML	200 ML			
TYPES/FAMILY (FILL OUT)	% COMPOSITION	% COMPOSITION	GENERA (FILL OUT)	% COMPOSITION	% COMPOSITION
CTENOPHORES (COMB-JELLIES)	100%	100%	BEROE		
			MNEMOPSIS		
SCYPHOZOA (TYPICAL JELLYFISH)	0%	0%	CHRYSAORA		
			AURELIA		

Pycnocline Calculation

A: _____ (surface conductivity) - _____ (bottom conductivity) = _____ (Conductivity)

B: Conductivity / (Depth in m) - 0.05 = _____ (Average Conductivity Change) * 2 = _____ (Pycnocline Threshold)

DEFINITIONS:

Upper Limit: The shallowest depth where change in conductivity ^{3} the Pycnocline Threshold Upper Depth

Lower Limit: The deepest depth where the change is conductivity ^{3} the Pycnocline Threshold Lower Depth

Note: No Pycnocline is: 1) Pycnocline Threshold < 0.5 mmho/cm, or
2) no depth interval > threshold value.

G. Mateja 1997

GENERAL INFORMATION	G. Mateja 1997
STATION: SBE5	SAMPLING DATE: 8/12/98
COLLECTED BY: SEL	

ENVIRONMENTAL DATA

DEPTH (M)	TEMPERATURE °C	SALINITY	SP. CONDUCT. (MMHO/CM)	DO (PPM)	pH	
1	32.00	18.11	29.55	4.86	7.12	
2	31.67	18.09	29.50	4.41	7.08	
3	31.49	18.09	29.50	4.49	7.11	
4	30.76	18.19	29.61	4.30	7.11	
5	30.52	18.33	29.80	4.28	7.12	
6						
7						
8						
9						
10						
11						
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25						
27						
29						
31						
33						
35						

Chief Scientist (Initials) : _____

Supervisor (Initials) : _____

12. Appendix 2 - **CBP MONITORING FIELD SUMMARY**

Cruise # :_____ Date : _____
Weather :_____ Day : 1 2 3 4 5
Field Chief/Crew :_____

Station	Arrive	Depart	Comments

AM PM

Pre-calibration(date/time): _____
Post-calibration (date/time) : _____

Cooler : _____ _____
Freezer : _____ _____

Post Calibration Values			
Conductivity	**S.V.**	**58.6 mmhos/cm**	
	I.V.	**mmhos/cm**	
pH	**S.V**	**7.00**	**10.00**
	I.V.		
D.O.	**S.V**	**mg/L**	
	I.V.	**mg/L**	

S.V.= Standard Value
I.V. = Instrument Value

Notes

_____
_____
_____
_____
_____

Temperature Checks