Standard Operating Procedure for Sampling Lake Michigan Lower Pelagic Foodchain for PCBs, Nonachlor, and Mercury

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Revision 1

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1.0 Zooplankton Sampling (>102 μm net, >500 μm net)

1.1 Equipment and Materials

Zooplankton net, 1 m diameter, 4 m long, 102 μm mesh Nitex netting
Zooplankton net, 1 m diameter, 4 m long, 500 urn mesh Nitex netting
PVC sample cups, 1000 mL volume, with 102 or 500 μm mesh
Winch
5 lb weight
Lake water hose
4 L glass bottle
1 L glass bottle
Poly pro funnel, 20 cm diameter
102 μm Nitex netting, 18" x 18", supported by poly pro large mesh strainer
Stainless steel kitchen strainer stainless spatula
Rectangular baking pan
Glass Qorpak 9 or 16 oz wide mouth jars
Spray bottle for filtered lake water
500 mL PFA teflon jar
PFA teflon spatula
Nalgene polycarbonate disposable analytical filter unit, 0.45 μm, 100 mL
60 mL PFA teflon jar
30 mL PFA teflon vial
10 mL autopipetter and disposable poly pro tips
Shurco vacuum pump
Nylon forceps

1.2 Preparation of materials and equipment

1.2.1 Net: wash by hosing down with lake water between stations and between casts.

1.2.2 Collection materials: rinse dewatering netting and strainer between uses with nanopure water.

1.2.3 Organics (PCBs and nonachlor): Qorpak jars are ashed at 450 C for minimum of 4 hours before use.

1.2.4 Mercury

1.2.4.1 All teflonware is acid-washed in concentrated nitric acid and rinsed with nanopure water and either dried under dust-free conditions or stored filled with 1% (v/v) HCl. It is stored in acid-washed polypro bags, double bagged.

1.2.4.2. All polypro and nylon is washed in 1.0 M nitric acid and rinsed in nano-pure water, and dried under dust free conditions.

1.2.4.3. Filtration units are used as received from vendor (come sealed).

1.3 Collection Procedure

1.3.1 Sample locations: pre-selected by GLNPO and LMMBS. Sites include three Biota Zones (Stations 110, 140, 180, 240, 280, 310, 340, and 380), two Master Stations (Stations 18 and 47), and Station 5 off of Chicago for organics and mercury. The order of sample collection is 47, 180, 140, 110, 280, 240, 18, 380, 340, 310, 5. All other Master Stations (8) are sampled for mercury analyses only, when possible.

1.3.2 Depth of tow: Vertical tows from near the bottom to surface (depth depends on water depth, time of day, and sea conditions) are done under standard net tow procedures from port side A-frame winch with the assistance of ship's crew. Net is attached to winch and safety line is attached to one of net cables. Cup safety line is attached to net rim.

1.3.3 Number of tows: is dependent on mass collected per tow. Several grams of wet weight of material are required for organics and mercury analyses; a few hundreds of mg of material are needed for mercury analyses only. Approximately 4-6 tows are typically needed for organics, 1-2 tows are needed for mercury only.

1.3.4 Isolating sample

1.3.4.1 Net is brought to just above the surface and lake water hose is used to wash down sides of net from outside so that material adhering to inside of net collects in bottom cup.

1.3.4.2 The cup is removed from the net and poured into the glass bottle (4 L bottle for 102 μm net, 1 L bottle for 500 μm net) via the funnel. Cup is rinsed and rinsate added to bottle. If another tow is required, the procedure is repeated.

1.3.5 Dewatering 102 μm sample: Contents of 4 L bottle are poured into netting held by strainer. Dewatered material is removed by spatula to appropriate container for either organics or mercury (see below).

1.3.6 Dewatering 500 μm sample: Contents of 1 L bottle are poured through stainless stell strainer, or into rectangular pan. Using forceps, sample is segregated into species-specific groups as much as possible. For instance, mysis are picked out with tweezers and removed to sample jar. Remainder of sample is removed to jar with spatula.

1.3.7 Apportionment of sample for organics and mercury analyses: Approximately 5% of the sample is reserved for mercury analysis. Only a minor fraction is required due to the difference in detection limits between PCBs and mercury. The aliquot for mercury is either taken directly from the bottle to the 500 mL PFA teflon jar by pouring prior to dewatering (if sample is highly concentrated), or after dewatering (if sample is not highly

concentrated). The remainder of the sample is transferred by spatula to the Qorpak jar for organics analysis.

1.3.8 Sample Processing and Handling

1.3.8.1 Mercury: Sample is isolated for analysis by filtration.

1.3.8.1.1 A mass determination is necessary to know the amount of sample being analyzed. This is accomplished by homogenizing sample by swirling container. A 10, 20, or 30 mL subsample is removed by pipet to a 30 mL PFA teflon vial and frozen for later dehydration and mass determination by standard gravimetric procedures.

1.3.8.1.2 Prior to sample filtration the filter is leached with 10 mL of 1% HCl. A known volume (10-100 mL) of sample suspension is filtered through the disposable analytical filtration unit. The filter is removed, placed in 60 mL PFA teflon jar, labeled according to labeling procedure (see Sampling QAPjP), double bagged, and frozen for transport and storage. If dewatered sample is used, it is resuspended in nanopure water and handled as above.

1.3.8.2 Organics: Dewatered sample is frozen in bulk.

Material in Qorpak jar is labeled according to labeling procedure (see Sampling QAPjP) and frozen for transport and storage. All appropriate tracking information is recorded in field notebooks. This includes the label i.d., the number of tows, the depth of the tow, and any species identification that has been made from microscopic analysis.

2.0 Phytoplankton Sampling (102 > P > 10 μm)

2.1 Equipment and Materials

Phytovibe with 10 μm Nitex netting and 700 mL PVC cup
Lake water hose
Two submersible pumping systems attached to nylon- 11 hose
102 μm Nitex net cover
500 mL PFA teflon jar
PFA teflon spatula
Nalgene polycarbonate disposable analytical filter unit, 0.8 μm, 100 mL
60 mL PFA teflon jar
30 mL PFA teflon vial
10 mL autopipetter and disposable poly pro tips
Shurco vacuum pump
Nylon forceps
1 L glass jar with graduated markings

10 mL glass graduated pipets
Pipet bulb
47 mm glass Millipore filtration apparatus (2)
47 mm plastic magnetic Nalgene filtration apparatus (2)
Stainless forceps
47 mm polycarbonate Nuclepore filters, pre-weighed
47 mm GF/F glass fiber filters, ashed
Plastic petri dishes
Aluminum foil, ashed
125 mm ceramic Buchner funnel
125 mm GF/F glass fiber filters, ashed

2.2. Preparation of Materials and Equipment

2.2.1 Phytovibe: is washed down with lake water between uses. If necessary, remove the 102 μm net cover from the end of the hoses and clean thoroughly with lake water, and replace. Cups are rinsed thoroughly with lake water. If flow through net during collection is restricted, net is removed between stations from phytovibe support and washed in the washing machine.

2.2.2 Organics

2.2.2.1 All glassware is wrapped in foil and ashed. All ashed materials are combusted at 450 C for a minimum of 4 hours.

2.2.2.2 Nuclepore filters are preweighed on a Satorius analytical balance in the laboratory, and individually stored in petri dishes for transport to and from the field.

2.2.2.3 The 47 mm GF/F glass fiber filters are wrapped in foil in packages of 9 and ashed. The 125 GF/F filters are individually wrapped in foil and ashed.

2.2.3 Mercury

2.2.3.1 All teflonware is acid-washed in concentrated nitric acid and rinsed with nano-pure water and either dried under dust-free conditions or stored filled with 1% HCl. It is stored in acid-washed polypro bags, and double bagged.

2.2.3.2 All prolypro and nylon is washed in 1.0 M nitric acid and rinsed in nano-pure water, and dried under dust free conditions.

2.2.3.3 Filtration units are used as received from vendor (come sealed).

2.3 Collection Procedure

2.3.1 Sample Locations: pre-selected by GLNPO and LMMBS. Sites include three Biota Zones (Stations 110, 140, 180, 240, 280, 310, 340, and 380), two Master Stations (Stations 18

and 47), and Station 5 off of Chicago for organics and mercury. The order of sample collection is 47, 180, 140, 110, 280, 240, 18, 380, 340, 310, 5. All other Master Stations (8) are sampled for mercury when possible.

2.3.2 Depth the collection (pumping) depth is chosen based on an interpretation of the temperature, fluorescence, and BA profiles from the SeaBird. The objective is to choose a depth that maximizes the occurrence (and hence collection) of Phytoplankton that are being grazed. This generally will be mid-epilimnion, or at the subthermocline chlorophyll maximum in stratified conditions.

2.3.3 Phytovibe operation

2.3.3.1 Once the ship is at anchor following the SeaBird and Rosette operations and with clearance from the Chief Scientist, pumps are placed at the sampling depth.

2.3.3.2 The outflow end is covered with a bag of 102 μm Nitex netting to remove large particles and secured with a hose clamp. The lines are flushed for a minimum of 15 minutes.

2.3.3.3 After flushing, the outflows are directed into the phytovibe, the vibrating motors turned on, and the pumps are allowed to pump for the duration of the time on station, or until sufficient mass (several grams of wet weight material for organics and mercury; several hundred mg material for mercury) is collected. Pumping rate is approximately 20-30 L/min. The netting at the end of the hose must be checked frequently to check for plugging. It is cleaned and/or replaced as necessary. The phytopvibes should be covered with a tarp if it is raining or if insects appear to be fouling the sample. Eight to ten hours of pumping time may be necessary. At several points during the pumping lake water should be used to rinse the sides of the net down by spraying the outside of the net.

2.3.4 Sample isolation: lake water is used to wash the material adhering to the net surface down into the cup by rinsing the outside of the net. When all the water has drained to below the top of the cup, the cup is removed to the extraction lab.

2.3.5 Apportionment of sample for organics and mercury analyses:
Approximately 5-10% of the sample is reserved for mercury analysis. Only a minor fraction is required due to the difference in detection limits between PCBs and mercury. The aliquot for mercury is taken directly from the cup to the 500 mL PFA teflon jar by pouring. This split is not quantitative, as the mass of sample analyzed for organics and mercury is determined separately for the different analyses. The remainder of the sample is transferred to the 1 L glass bottle for organics analysis.

2.3.6 Sample Processing and Handling

2.3.6.1 Mercury: Sample is isolated for analysis by filtration.

2.3.6.1.1 A mass determination is necessary to know the amount of sample being analyzed. This is accomplished by homogenizing sample by swirling the container. A 10, 20, 30 mL subsample is removed by pipet to a 30 mL PFA teflon vial and frozen for later dehydration and mass determination by standard gravimetric

procedures.

2.3.6.1.2 Prior to sample filtration the filter is leached with IO mL of 1 % HCl. A known volume (10- 100 mL) of sample suspension is filtered through the disposable analytical filtration unit. The filter is removed, placed in 60 mL PFA teflon jar, labeled according to labeling procedure, double bagged, and frozen for transport and storage.

2.3.6.2 Organics: The sample is diluted to a known volume, subsampled for mass and carbon determinations, and collected on a filter for analysis.

2.3.6.2.1 Subsampling: This is accomplished by diluting the sample in the 1 L bottle to a known volume with filtered lake water.

2.3.6.2.1.1 Dry mass: A known volume (1 - 2 mL) is removed in duplicate by pipet for filtering through a pre-weighed 1.0 μm 47 mm Nuclepore filter for dry mass determination by standard gravimetric procedures. The filter reservoir is rinsed with a small amount of nanopure water, and the filter folded in quarters and placed back in the petri dish for transport and storage. All volumes and pertinent information is recorded in the Mass field notebook and master file. This includes: filter i.d. number, tare weight in mg (previously recorded in notebook in lab), sample label i.d., volume of sample filtered.

2.3.6.2.1.2 Organic Carbon: A known volume (1 - 2 mL) is removed in duplicate by pipet for filtering through an ashed 47 mm. GF/F filter for particulate organic carbon (POC) determination. The filter reservoir is rinsed with a small amount of nanopure water. The filter is folded in half, wrapped in ashed foil, labeled, and the wrapped filters placed in labeled ziplock bags which are frozen for transport and storage. All pertinent information is recorded in the POC field notebook and master file. This includes: sample label i.d., and volume filtered.

2.3.6.2.2 Processing: The remainder of the sample is filtered through a 125 mm GF/F glass fiber filter in a Buchner funnel to isolate the Phytoplankton from suspension. The filter :is placed in the Buchner funnel, wetted with nanopure water, and vacuum applied. The bottle contents are then carefully poured in. The

bottle is rinsed twice with filtered lake water and the rinsate passed through the filter. The filter is folded in quarters, wrapped in ashed foil, labeled (see Sampling QAPjP), placed in labeled ziplock bag, and frozen for transport and storage. If any residual sample is on the inner rim of the Buchner funnel, the rim is wiped with a wetted kimwipe, and the kimwipe added to the foil package within the ziplock. This is analyzed along with the filter. Pertinent information to be recorded includes: sample label i.d., approximately time the phytovibe was turned on and off, depth of the water that was sampled, volume the organic sample was diluted to, volumes of subsamples removed for mass and carbon determinations.

3.0 Detrital Fraction Sampling (Organic Analytes Only)

3.1 Materials and Equipment

293 mm stainless filtration apparatus
280 mm stainless stacked filtration apparatus
Peristaltic pumps .
½" od polyethylene tubing
293 mm GF/F glass fiber filters, ashed
280 mm 102 μm nitex netting
280 mm 10 μm nitex netting
Teflon wash bottle with nanopure water
Teflon wash bottle with methanol
Large kimwipes
Large stainless steel forceps (2)
Ziplock bags

3.2 Preparation of Materials and Equipment

3.2.1 The filtration apparati are wiped clean with a kimwipe wetted with methanol, and rinsed with nanopure water between samples.

3.2.2 Nitex netting is rinsed with nanopure water.

3.3 Collection Procedure

3.3.1 An ashed 293 mm GF/F glass fiber filter is placed on the filter holder with forceps and wetted with nanopure water. The top of the filter head is replaced and secured.

3.3.2 The 10 μm nitex net is placed on a stainless steel screen support on the bottom-most layer of the stacked filter system, the next stage is added, and the 100 μm net is placed on the stainless steel screen support. The top of the system is then added and secured. The system is slowly filled with nanopure water from the bottom (reverse direction from sample collection) so that undue pressure from the incoming sample does not rupture or

break the seal of the 10 μm net. The system is charged by attaching the outflow hose from the bottom of the filtration system to the outflow of the nanopure water.

3.3.3 The polyethylene tubing is replaced at the beginning of the sampling cruise (prior to Station 47), after the first Biota Zone (after Station 110), and after the end of the third Biota Zone (Station 310). This is to prevent contamination from a more contaminated site to a less contaminated site by desorption of the target analytes from the tubing.

3.3.4 The submersible pump is placed at the appropriate sample collection depth (see 2.0 Phytoplankton Sampling, above) and the lines are flushed (up to the peristaltic pump) for approximately 30 minutes. The pump and plumbing from the pump to the extraction lab are provided by the ship. The plumbing within the extraction lab is provided by the University of Minnesota.

3.3.5 The water flow is as follows: water is drawn by submersible pump through nylon- 11 line to the deck of the ship and flows to the outer door of the extraction lab. A T in the line allows for some of the water to be drawn into the lab, with the remainder returned to the lake. Water is drawn by peristaltic pump through polyethylene tubing from the T, delivered to the top of the stacked filtration apparatus, and the outflow from the apparatus is drawn by a second head of the same peristaltic pump and delivered to the top of the 293 mm filter head. The outflow is collected in teflon lined stainless steel kettles for dissolved contaminant extraction, or discharged overboard. Water must be pumped to and from the stacked filtration apparatus to minimize pressure on the 10 μm nitex layer.

3.3.6 The pumps are turned on, and the time recorded in the field notebook. The pump setting should be approximately 4. Air is removed from the system by holding the outflow closed with a finger and opening the pressure release valve at the top of the 293 mm filter head until water comes out. The flow rate of the water through the glass fiber filter is determined at the beginning, and every hour until filtering ends, unless a filter is changed in which case the flow rate is determined a minimum of at the beginning of the filtering and at the end just prior to changing the filter. Flow rate is determined by collecting exactly 1 L of water in a polypropylene graduated cylinder and noting the time on a stopwatch. The flow rate should be 4-5 L/min. When time permits duplicate flow rate measurements should be taken at any given time point. The setting should not require adjustments during a cruise. If changes are made, flow rate must be determined at the time of change and the time the setting was changed must be recorded to determine the volume of water processed with sufficient accuracy.

3.3.7 When pressure on the 293 filter head exceeds 5-6 psi, the glass fiber filter should be changed. This is to prevent significant lysis of cells in the detrital fraction. To change a filter, the peristaltic pump is stopped and the time recorded. The outflow from the stacked filtration apparatus is disconnected from the peristaltic pump and directed to waste, and the pumps turned on to remove water from the 293 mm filter head (i.e. air is being pumped through the 293 mm filter head). The peristaltic pump is turned off, and the filter head is dissassembled, the filter is folded in quarters using the large forceps, and wrapped in ashed foil. It is labeled, placed in a ziplock bag, and frozen for transport and storage. All filters for one sample are stored together in one ziplock bag. The order of the filter is indicated on the individual filter label. All filters will be analyzed together as one sample. The filter head is wiped clean with a kimwipe wetted with nanopure water, and a new filter installed as described above. The outflow from the stacked filtration apparatus is reconnected to

the peristaltic pump, and the pumps restarted. The time of restart is noted, and flow rate determined.

3.3.8 When sufficient mass of the detrital fraction has been collected (approximately 1000 L of water, or 4 x 293 mm filters) the peristaltic pump is turned off and the time noted. The 293 mm filter is removed as described above. The stacked filtration apparatus is dissassembled, and the netting removed, washed thoroughly in nanopure water, and examined for rips or -holes before being replaced for the next sample. Total water volume for this sample is calculated as:

*volume = [(rate, L/min) * (min)] - (volume of subsamnples removed for filtering, L)*

3.3.9 Subsampling

3.3.9.1 Dry Mass: A known volume (150-250 mL) is removed through the valve in the water stream on the 293 mm filter head just prior to the 293 mm filter. Water is filtered through a pre-weighed 0.4 μm 47 mm Nuclepore filter for dry mass determination by standard gravimetric procedures. The filter reservoir is rinsed with a small amount of nanopure water, and the filter folded in quarters and placed back in the petri dish for transport and storage. All mass determinations are done in duplicate. All volumes and pertinent information is recorded in the Mass field notebook and master file. This includes: filter i.d. number, tare weight in mg (previously recorded in notebook in lab), sample label i.d., and volume of sample filtered, and volume removed for filtering.

3.3.9.2 Organic Carbon: A known volume (1.5 - 2 L) is removed by dispensing from the valve in the water stream on the 293 nim filter head just prior to the 293 mm filter. Water is filtered through a 47 mm GF/F filter for particulate organic carbon (POC) determination. The filter reservoir is rinsed with a small amount of nanopure water. The filter is folded in half, wrapped in ashed foil, labeled, and the wrapped filters placed in labeled ziplock bags which are frozen for transport and storage. All POC filtrations are done in duplicate. All pertinent information is recorded in the POC field notebook and master file. This includes: sample label i.d., volume filtered, and volume removed for filtering.

4.0 Transport and Storage

4.1 Sample Packing: All frozen samples are removed from the ship's freezers and immediately packed in coolers with frozen blue ice just prior to transport. XAD-2 columns are stored in refrigerators at 4 C. They are also packed in coolers and kept cold with blue ice during transport. Coolers are taped shut to prevent inadvertent opening during transport.

4.2 Sample transport: Samples are transported in coolers by University of Minnesota personnel. Samples will remain either directly in the custody of the personnel performing transport, or in the possession of commercial air carriers if the personnel travel by air.

4.3 Sample Logging: All samples are logged out of ship's storage at the time they are packed into

coolers, and again at arrival at the PIs' laboratories at the University of Minnesota as they are placed into storage. Sample logs will note sample number, date of each sample transfer, initials of personnel responsible for custody during each stage of transport, and final storage location of each sample iii the PIs' laboratories. Examples of tracking forms are shown in Figures 1 and 2.

4.4 Sample Custody: The sample log indicates the personnel responsible for sample custody during transport. Samples will remain in the custody of ship's personnel while in storage onboard ship, the University of Minnesota personnel during transport, and their respective PI once checked into the PI's laboratory.

4.5 Sample Storage in the Laboratory: Labeled samples will be stored in freezers or refrigerators located in the PI's laboratories. All labs are locked except when in use.

	CRUISE:								
		date/initials		date	date	date	date	date	date
	Sample i.d.	from ship	to UMN	extracted	cleaned	GC-ECD	NCI	baselines	final quant.
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
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25									
26									
27									
28									
29									
30									

Sample id:
Date collected:
Time collected:
Initials of individual collecting sample:
Time of storage:
Date of removal from storage:
Initials of transport personnel:
Date of arrival at laboratory:
Location of storage:
Date of processing
    Digestion:
    Dehydration:
Date of analysis
    Hg measurement:
    Weighing:
Standard curve identifier:

Figure 2. Example tracking form for Hg

5.0 Dissolved Fraction Sampling (Organic Analytes Only)

5.1 Materials and Equipment

75 L teflon lined stainless steel kettles with stainless steel lids (2)
Small peristaltic pump
Teflon and tygon tubing, 3/8 " id
Glass columns, 3x30 cm, packed with cleaned XAD-2 resin
Wash bottle with methanol
Wash bottle with nanopure water
Kimwipes
Strap wrench
100 mL polycarbonate bottle

5.2 Preparation of Material and Equipment

5.2.1 XAD-2 resin: The resin is pre-cleaned in the laboratory by sequential Sohxlet extraction, and packed in individual extraction columns for transport to and from the field. It is cleaned in large batch quantities by extracting for 24 hours with methanol, followed by 24 hours with acetone, followed by 24 hours with hexane, followed by 24 hours with dichloromethane. It is then extracted with the same solvents in reverse order for 4 hours each, and then washed thoroughly with nanopure water. It is stored in amber bottles under water until the columns are packed.

5.2.2 Resin Columns: The glass columns are ashed at 450 C for a minimum of 4 hours. A teflon end cap with outflow hole is placed on one end, and a plug of ashed glass wool is added. The outflow is blocked, and resin in nanopure water is poured into the top of the column through a funnel. The water is allowed to drain from the outflow as necessary to allow the resin to settle and to reduce the volume of water in the column, while never allowing the level of the water to fall below the resin. The columns are filled to approximately 2/3 their capacity (approximately 150 mL resin and water), a glass wool plug added to secure the resin in place, and the columns are topped with nanopure water and end caps secured on either end. The columns are wrapped in foil, wrapped in bubblewrap, and stored in a cooler for transport and storage.

5.2.2 Stainless steel kettles: The kettles are wiped thoroughly with kimwipes and methanol, followed by a thorough rinse with nanopure water. The lids are taped on to prevent contamination before use.

5.2.3 The polycarbonate bottles are for dissolved organic carbon samples. They are washed with soap and water, rinsed with tap water followed by nanopure water, soaked in 2% nitric acid for 24 hours, rinsed with nanopure water, soaked for 4 hours with nanopure water, and filled with nanopure water until use.

5.3 Sample Collection

5.3.1 The outflow from the 293 mm filter is directed to the kettles. The foil is removed from two XAD columns. The endcap of the outflow end of each of the XAD columns is replaced with an endcap with a quick-connect fitting to teflon tubing which flows to the small peristaltic pump. The column and tubing are wiped a kimwipe wetted with methanol, followed by a wipe using nanopure water. The inflow endcap is replaced with an endcap with a hole, any air is relieved with nanopure water, and using a finger to hold this closed the column is immersed in the water and the finger released. The peristaltic pump is started at the same time, and the time noted. A setting of about 3 should produce the desired flow rate of 300 mL/min. The outflow of the peristaltic pump is directed overboard.

5.3.2 Flow rates are determined at the beginning, at 30 minutes, at 60 minutes, and then hourly until the extraction is complete. Flow rate is determined by filling a 250 mL graduated cylinder and noting the time with a stopwatch. The outflows from each column are both monitored. Time of measurement, and flow rate, are recorded in the XAD field notebook. Total volume in L is a volume-weighted sum of minutes pumped times the flow rate for that time period in L/minute. A minimum of 200 L is extracted; 300 L is desirable. Thus several volumes of the kettles are processed. Particulate filtering must occur long enough to allow for the generation of a sufficient volume of water to complete the dissolved phase extraction.

5.3.3 When sufficient volumes of water have been passed through the resin, the peristaltic pump is turned off and the time recorded. The resin column endcaps are replaced with the storage endcaps, the sample is labeled, the foil and bubblepack are replaced, and the columns are placed in the refrigerator until transit back to the laboratory.

5.3.4 Subsamples: Samples for the measurement of dissolved organic carbon are collected from the XAD inflow. A 100 mL polycarbonate bottle is rinsed with the sample water, filled approximately halfway, labeled, and frozen for transport and storage.