CONTROLLED DOCUMENT

TS-IOG-003
Subsampling Water Samples in the Field

Revision 1.1

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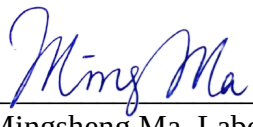

Dr. Mingsheng Ma, Laboratory Manager — 03/11/2016 Date

Subsampling Water Samples in the Field

1. Purpose:

In situations where whole water samples cannot be submitted to the Biogeochemical Analytical Service Laboratory within 48 hours, either by courier or in person, the field person can subsample in the field. The sample may need to be subsampled into unfiltered, filtered, filtered/acidified or filtered/frozen depending on the analyses required. This procedure gives the subsampling steps that can be done in the field and the storage conditions for each subsample.

2. Summary of procedure:

The volume required for each subsample will vary depending on the parameters requested. An unfiltered subsample is poured for TP, TN, TKN, pH, alkalinity, NFR, conductivity, turbidity and silica. A 0.45µm cellulose acetate filter is used for subsampling of TDP, TDN, DOC, DIC, SO4, Cl, absorbance, color, major ions, trace metals, and TDS. For NO_2/NO_3 and NH_4, and SRP, 12mL of sample is filtered using a syringe filter with a 0.45µm cellulose acetate filter attached, this filtrate is then frozen or shipped to the lab within 48 hours if freezing cannot be done. CHN and chlorophyll a are collected on filters, whereas chlorophyll a filters will be frozen.

3. Definitions:

3.1 TP: Total Phosphorus

3.2 TN: Total Nitrogen

3.3 TKN: Total Kjeldahl Nitrogen

3.4 TDN: Total Dissolved Nitrogen

3.5 TDP: Total Dissolved Phosphorus

3.6 SRP: Soluble Reactive Phosphorus

3.7 DOC: Dissolved Organic Carbon

3.8 DIC: Dissolved Inorganic Carbon

3.9 SO4: Sulfate

3.10 Cl: Chloride

3.11 MI: Major Ions

3.12 TM: Trace Metals

3.13 TDS: Total Dissolved Solids

3.14 NO_3: Nitrate

3.15 NO_2: Nitrite

3.21 NH_3: Ammonia

3.22 NFR: Non Filterable Residue

3.23 CHN: Carbon, hydrogen, nitrogen

3.24 DI Water : Deionized water

4. Apparatus and Equipment:

4.1 Filtration tower:

- 4.1.1 Erlenmeyer filter flask
- 4.1.2 Filter tower (cup and frit)
- 4.1.3 Conical filter tower (CHN)

4.2 Hand pump

4.3 Filters

4.4 Ethanol prewashed

4.5 47mm 0.7µm Thermo Scientific Target GMF syringe filters

4.6 30mm 0.7µm Thermo Scientific Target GMF syringe filters

4.7 Muffled 25mm 0.7µm Whatman GF/F filters

4.8 20mL Luer-Lok tip syringes/60mL Luer-Lok tip syringes

4.9 0.45µm cellulose acetate syringe filters

4.10 DI water rinsed 0.45µm cellulose acetate filters

4.11 70mL plug Corning culture flask

4.12 15mL centrifuge tubes

4.13 50mL centrifuge Tubes

4.14 Wash bottles (DI Water)

4.15 Amber or White Nalgene bottles

4.16 10x10 storage box

5 Reagents

5.1 18% Nitric Acid (3:1 ratio of Trace Grade Nitric Acid and Deionized Water)

5.2 DI Water

6. Personal qualifications:

Subsampling in the field is to be performed by appropriately trained field people.

7. Procedure:

Please subsample the water sample in the order of this section.

7.1 NO_3NO_2/NH_3/SRP/Stable Isotope:

7.1.1 Label a 15mL centrifuge tube with the sample information and "N/A" if both NO_3NO_2 and NH_3 are requested (do not label the cap). If only NO_3NO_2 or NH_3 is requested, label the tube with "N" or "A" respectively.

7.1.2 Label a 15mL centrifuge tube "SRP" (do not label the cap) if SRP is requested.

7.1.3 If Stable Isotope is also requested, label a 2mL glass vial with O/H, together with the site information.

7.1.4 Open the syringe package (20mL if only N/A is requested, 60mL if SRP is also requested) and pull the plunger out of the syringe. Lay the plunger in a clean, dry place.

7.1.5 Screw on a 0.45µm cellulose acetate syringe filter.

7.1.6 Invert the sample 10 times.

7.1.7 Rinse the syringe and the plunger with a small portion of sample.

7.1.8 Fill the syringe by pulling the plunger up.

7.1.9 Push 5mL of subsample through the filter and discard the filtrate. Use another small portion (around 5mL) of filtrate to rinse the centrifuge tubes.

7.1.10 For N/A, fill the centrifuge tube with filtrate up to 12mL.

7.1.11 If samples are very turbid and difficult to filter, attach a 30mm 0.7µm Thermo Scientific Target GMF syringe filter as a pre-filter prior to the 0.45µm cellulose acetate filter in the order of "syringe - 0.7µm GF/F filter - 0.45µm AC filter".

7.1.12 For SRP subsample, fill the centrifuge tube with filtrate up to 12mL.

7.1.13 For Stable Isotope, fill the filtrate up to the top of the container with no headspace and cap the screw cap tightly to avoid evaporation. The isotope samples can be stored in a 10x10 storage box.

7.1.14 Store the NO_2/NO_3 and NH_4, and SRP subsamples in Ziploc bag at -20ºC or ship to the lab within 48 hours at 4ºC. Do not freeze the centrifuge tubes with the foam racks which could cause tube crack.

7.1.15 Store the Stable Isotope samples at 4ºC.

7.2 TP, TN, pH, alkalinity, NFR, TDS, conductivity, turbidity and silica:

7.2.1 Determine the total volume required using the Chain of Custody or consult with lab staff for the minimum volume required (section 9.0).

7.2.2 Invert the sample 10 times.

7.2.3 Rinse the appropriate sized Nalgene bottle 3 times with a portion of the sample.

7.2.4 Fill the Nalgene bottle with unfiltered sample.

7.2.5 Store the subsample at 4ºC.

7.3 Chlorophyll a:

7.3.1 Chlorophyll a should be subsampled in dim light environment.

7.3.2 Label a petri dish with the sample information and “chl a”.

7.3.3 Place an ethanol prewashed 47 mm 0.7µm GF/F filter on the filter tower.

7.3.4 Invert the sample 10 times.

7.3.5 Depending on how turbid the sample will be, measure 30mL-100mL of sample into a graduated cylinder. Pour a small portion of the subsample through the filter to rinse the inside of the filter flask and empty filtrate.

7.3.6 Pour the rest of the subsample through the filter. If there is no color observed on the filter, continue filtering a **known volume** of sample through the filter until color is observed. Up to 1000 mL or more volume may be required for very clear samples.

7.3.7 The filtrate is transferred from the filter flask and may be used for other filtered parameters.

7.3.8 Replace the filter tower on the flask and rinse the graduated cylinder and the filter tower 3 times with DDW.

7.3.9 Fold the filter in half with the color sided on the inside and fold the filter in half again.

7.3.10 Place the folded filter in the petri dish and label the petri dish with the **volume filtered**.

7.3.11 A duplicate is required for each sample. Repeat section 7.4.1 through 7.4.9, filtering the same volume of sample.

7.3.12 Place the duplicate in the same petri dish as 7.4.10.

7.3.13 Wrap the petri dish with foil.

7.3.14 Store the chlorophyll filters between -10ºC to -20ºC.

7.4 CHN:

7.4.1 Label a petri dish with the sample information and "CHN".

7.4.2 Place a muffled 25mm 0.7µm GF/F filter on the conical filter tower.

7.4.3 Measure a volume of sample into a graduated cylinder. The volume of sample used is dependant on the amount of particulate in the sample. Continue filtering a known volume of sample through the filter until color is observed.

7.4.4 Rinse the tower, graduated cylinder and filter 3 times with DI Water.

7.4.5 Place the filter in a petri dish, particulate side up and label the petri dish with the **volume filtered**.

7.4.6 If possible, dry the filters in an oven at 50ºC for one week and store the filters in a desiccator until they can be submitted. If drying the filters is not possible, store the CHN filters at ambient temperature.

7.5 TDN, TDP, DOC, DIC, SO4, Cl, absorbance, color, major ions, and trace metals:

7.5.1 Determine the total volume required for parameters using the Chain of Custody (section 9.0) or consult with lab staff for the minimum volume required.

7.5.2 If parameters requested require a large sample volume (>60mL), use the filter tower method for filtration. If parameters requested require a small sample volume (<60mL), use the syringe filter method for better efficiency.

7.5.3 Major ions and trace metals should be acidified in the field by filling a 15mL centrifuge tube up to 12mL of filtered sample and adding 4 drops of 18% nitric acid using a disposable pipette.

7.5.4 Store subsamples at 4ºC

7.5.5 Filtration using Filter Tower:

7.5.5.1 Place a DI water prewashed 0.45µm cellulose acetate filter on the filter tower.

7.5.5.2 Invert the sample 10 times.

7.5.5.3 Pour a small portion of sample through the filter, remove the filter tower, swirl the filter flask and discard the filtrate.

7.5.5.4 Place the filter tower back on the filter flask and filter the additional volume of sample required for the filtered parameters.

7.5.5.5 Rinse the sample bottle 3 times with the filtrate and discard.

7.5.5.6 Fill an appropriate sized sample bottle with filtrate.

7.5.6 Filtration using Syringe Filter:

7.5.6.1 To collect filtered samples, use a 15mL centrifuge tube if only one parameter is requested. For multiple parameters, use a 50mL centrifuge tube.

7.5.6.2 Label 15mL or 50mL centrifuge tubes with the sample information and parameter(s) requested (do not label the cap).

7.5.6.3 Open the syringe package and pull the plunger out of the syringe. Lay the plunger in a clean, dry place.

7.5.6.4 Screw on a 0.45µm cellulose acetate syringe filter.

7.5.6.5 Invert the sample 10 times.

7.5.6.6 Rinse the syringe and the plunger with a small portion of sample.

7.5.6.7 Fill the syringe by pulling the plunger up.

7.5.6.8 Push 5mL of subsample through the filter and discard the filtrate. Use another small portion (around 5mL) of filtrate to rinse the centrifuge tubes.

7.5.6.9 Fill the 15mL or 50mL centrifuge tubes with the filtrate.

7.5.6.10 If samples are very turbid and difficult to filter, attach a 30mm 0.7µm Thermo Scientific Target GMF syringe filter as a pre-filter prior to the 0.45µm cellulose acetate filter in the order of "syringe - 0.7µm GF/F filter - 0.45µm AC filter".

8. Quality Control:

8.1 Field blank and field duplicate are recommended to monitor the quality of the sub-sampling process. Field blank and duplicate should be treated as a sample during sub-sampling.

8.2 Field blank – field blank should be added into each project with a separate sample ID number. The frequency of field blank added is 1 in every 10 samples. Field blank can be obtained by getting a bottle of deionized water from the lab and bring it to the field.

8.3 Field duplicate – field duplicate is added into each project with a separate sample ID number. The frequency of field duplicate added is 1 in every 20 samples. Duplicate is obtained by sub-sampling the same sample twice.

9. Appendix: Chain of Custody can be downloaded from the BASL website at

http://www.biology.ualberta.ca/basl/

10. Revision History:

Revision No.	Version Date	Description of Change	Written By	Approval By
1.0	August 15, 2014	Initial Release	Alvin Kwan	Mingsheng Ma
1.1	March 11, 2016	• Update all field subsampling procedure in section 7.0.	Maya Abou-Ghanem	Mingsheng Ma