

P-006
Subsampling Water Samples in the Field

Revision 1.0

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Subsampling Water Samples in the Field

1. Introduction and Scope:

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 0.7um GF/F filtered subsample is taken for TDP, TDN, DOC, DIC, SO₄, Cl, absorbance, color, Na, K, Ca, Mg, Fe and TDS. 12mL of the 0.7um GF/F filtrate is frozen for SRP. 12mL of sample is filtered using a syringe filter and acidified with sulfuric acid for NO₃NO₂ and NH₄. 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 Na: Sodium
- 3.12 K: Potassium
- 3.13 Ca: Calcium
- 3.14 Mg: Magnesium
- 3.15 Fe: Iron
- 3.16 TDS: Total Dissolved Solids

- 3.17 NO₃: Nitrate
- 3.18 NO₂: Nitrite
- 3.19 NH₄: Ammonium
- 3.20 NFR: Non Filterable Residue
- 3.21 CHN: Carbon, hydrogen, nitrogen

4. Equipment/Chemicals:

- 4.1 Equipment:
 - 4.1.1 Filtration tower
 - 4.1.1.1 Erlenmeyer filter flask
 - 4.1.1.2 Filter tower (cup and frit)
 - 4.1.1.3 Conical filter tower (CHN)
 - 4.1.2 Hand pump
 - 4.1.3 Filters
 - 4.1.3.1 DDW prewashed 47mm 0.7um Whatman GF/F filters
 - 4.1.3.2 Ethanol prewashed 47mm 0.7um Whatman GF/F filters
 - 4.1.3.3 47mm 0.7um Whatman GF/F filters
 - 4.1.3.4 Muffled 25mm 0.7um Whatman GF/F filters
 - 4.1.4 20mL Luer-Lok tip syringes/60mL Luer-Lok tip syringes
 - 4.1.5 30mm 0.7um Target GF/F syringe filters
 - 4.1.6 70mL plug Corning culture flask
 - 4.1.7 15mL centrifuge tubes
 - 4.1.8 Wash bottles (DDW)
 - 4.1.9 Amber or White Nalgene bottles (volumes based on the analyses required)

5. Personal qualifications:

Sub-sampling can be performed by properly trained field people.

6. Procedure:

As stated in the Quality Management System, the order in which a whole water sample is to be sub-sampled is nitrogen first and then the remaining parameters.

6.1 NO₃NO₂/NH₄/DIC/SRP/Stable Isotope:

- 6.1.1 Label a 15mL centrifuge tube with the sample information (do not label the cap) and “N/A”. If only NO₃NO₂ or NH₄ is requested, label the tube with “N” or “A” respectively.
 - 6.1.2 Label two 15mL centrifuge tubes with DIC or SRP label on it if both tests are requested as well.
 - 6.1.3 If Stable Isotope also requested, label two 2mL glass vials with O/H, together with the site information on it.
 - 6.1.4 Open the syringe package (20mL if only N/A is requested, 60mL if DIC/SRP are also requested) and pull the plunger out of the syringe. Lay the plunger in a clean, dry place.
 - 6.1.5 Screw on a 30mm GF/F syringe filter.
 - 6.1.6 Invert the sample 10 times.
 - 6.1.7 Rinse the syringe and the plunger with a small portion of sample.
 - 6.1.8 Fill the syringe.
 - 6.1.9 Push 5-10mL of subsample through the filter and discard the filtrate. Use another small portion (around 5-10mL) of filtrate to rinse the centrifuge tubes.
 - 6.1.10 For A/N, fill the centrifuge tube with filtrate up to 12mL.
 - 6.1.11 For SRP subsample, fill the centrifuge tube with filtrate up to 12mL.
 - 6.1.12 For DIC subsample, fill the centrifuge tube with filtrate up to the top with no headspace.
 - 6.1.13 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.
 - 6.1.14 For storage of the NO₃NO₂, NH₄ subsamples:
 - 6.1.13.1 Add 20uL H₂SO₄ into 12ml of filtrate if freezing is impossible in the field.
 - 6.1.13.2 Store samples at 4°C.
 - 6.1.15 Store the DIC subsamples at 4°C.
 - 6.1.16 Store the SRP subsamples at -20°C or ship to the lab within 48 hours at room temperature.
 - 6.1.17 Store the Stable Isotope samples at 4°C.
- 6.2 **TN/TKN:**
- 6.2.1 Label a 70mL culture flask with the sample information and “TN” or “TKN” if both analyses are requested.
 - 6.2.2 Invert the sample 10 times.
 - 6.2.3 Rinse the culture flask with a small portion of sample.
 - 6.2.4 Fill the culture flask, leaving a small headspace, and cap the flask.

- 6.2.5 Store the TN/TKN subsamples at 4°C.
- 6.3 **TP, pH, alkalinity, NFR, conductivity, turbidity and silica:**
- 6.3.1 Determine the total volume required using the Chain of Custody (section 8, appendix 1).
- 6.3.2 Invert the sample 10 times.
- 6.3.3 Rinse the appropriate sized Nalgene bottle 3 times with the sample.
- 6.3.4 Fill the Nalgene bottle with unfiltered sample.
- 6.4 **Chlorophyll a:**
- 6.4.1 Chlorophyll a should be subsampled in dim light environment.
- 6.4.2 Label a petri dish with the sample information and “chl a”.
- 6.4.3 Place an ethanol prewashed 0.7um GF/F filter on the filter tower.
- 6.4.4 Invert the sample 10 times.
- 6.4.5 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 and discard from the filter flask.
- 6.4.6 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 500mL may be required for very clear samples.
- 6.4.7 The filtrate is transferred from the filter flask and may be used for other filtered parameters.
- 6.4.8 Replace the filter tower on the flask and rinse the graduated cylinder and the filter tower 3 times with DDW.
- 6.4.9 Fold the filter in half with the color sided on the inside and fold the filter in half again.
- 6.4.10 Place the folded filter in the petri dish and label the petri dish with the **volume filtered**.
- 6.4.11 A duplicate is required for each sample. Repeat section 6.4.1 through 6.4.9, filtering the same volume of sample.
- 6.4.12 Place the duplicate in the same petri dish as 6.4.10.
- 6.4.13 Wrap the petri dish with foil.
- 6.4.14 Store the chlorophyll filters between -10°C to -20°C.
- 6.5 **CHN:**
- 6.5.1 Label a petri dish with the sample information and “CHN”.
- 6.5.2 Place a muffled 25mm 0.7um GF/F filter on the conical filter tower.
- 6.5.3 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.

- 6.5.4 Rinse the tower, graduated cylinder and filter 3 times with DDW.
- 6.5.5 Place the filter in a petri dish, particulate side up and label the petri dish with the **volume filtered**.
- 6.5.6 If possible, dry the filters in an oven at 50°C 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.
- 6.6 **TDN, TDP, DOC, SO₄, Cl, absorbance, color, Na, K, Ca, Mg, Fe and TDS:**
 - 6.6.1 Determine the total volume required using the Project Chain of Custody Field Sheet (section 7, appendix 1).
 - 6.6.2 Place a DDW prewashed 47mm 0.7um GF/F filter on the filter tower.
 - 6.6.3 Invert the sample 10 times.
 - 6.6.4 Pour a small portion of sample through the filter, remove the filter tower, swirl the filter flask and discard the filtrate.
 - 6.6.5 Place the filter tower back on the filter flask and filter the additional volume of sample required for the filtered parameters.
 - 6.6.6 Rinse the Nalgene bottle 3 times with the filtrate and discard.
 - 6.6.7 Fill an appropriate sized Nalgene bottle with filtrate from 6.6.5.

7. Quality Control:

- 7.1 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.
- 7.2 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.
- 7.3 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.

8. Appendix 1: Chain of Custody

Website: <http://www.biology.ualberta.ca/BASL>