

MEASURING AND MODELLING THE DYNAMIC RESPONSE
OF REMOTE MOUNTAIN LAKE ECOSYSTEMS TO
ENVIRONMENTAL CHANGE

A programme of **MO**untain **LA**ke **R**esearch

MOLAR

SURFACE WATER

SAMPLING AND ANALYSIS PROTOCOL

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Surface Water - Sampling and analysis protocol

1. Major ions and nutrients WP1, 2 and 3

The standard water samples are taken as surface grab samples from the outlet of the lake. Variability in lake properties has an annual cycle, and to study in more detail this cycle the following (minimum) sampling frequency programme will be followed:

Winter (Nov.-Dec.-Jan.-Feb.-March)	2 samples
Spring (April/May-May/June)	8 samples
<u>Summer/Autumn (May/June-Oct.)</u>	<u>4 samples</u>
Total number of samples pr. site	<u>14 samples</u>

Prevention of sample contamination or sample changes during storage may be critical in obtaining accurate measurements for these water samples of very low total ionic strength. All containers used for sample collection or storage must be free of any important quantity of the determinands in relation to the lowest concentration to be measured. The containers must also be of material that will neither absorb/adsorb nor release measurable quantities of the determinand.

2. Heavy metal determination WP2

Four samples from the fish containing lakes in WP 2 (Ø. Neådalsvatn, Redo, Gossenköllesee and Jorisee), will be analysed at NILU for Hg, Cd, Pb, Cu, Co and Zn. The sample bottles will be mailed from NILU to the different participants on agreed and due time before the sampling. The procedures for cleaning and preparation of the sample bottles will be as described under 4.4 in this part of the manual. The following bottles will be sent:

- For Hg glass bottle specially washed, preserving agent added (acidified water)
- For Cd + Pb plastic bottle specially washed for heavy metal analysis preserving agent added (acidified water)

All water samples should be unfiltered. The samples will be taken from the lake outlet in September/October, February, May and July to cover seasonal variations.

The sample bottles should be returned to NILU and marked clearly:

MOLAR - name of lake/site
Torunn Berg/Kjetil Tørseth
Norwegian Institute for Air Research (NILU)
P.O. Box 100
2007 Kjeller, NORWAY

Phone + 47 63 89 80 00
Telefax + 47 63 89 80 50

3. Heavy Metals (Speciation)

3.1 Introduction

Lake water samples are to be collected for trace metal (lead, copper) speciation and total metal (lead, copper, zinc, iron, chromium) analyses. Metal concentrations in remote lakes are expected to be low (much lower than in rain water), so clean

procedures should be followed to prevent sample contamination with metals from sampling gear or hands.

Samples should be collected at several depths from the water column of the lake: typically at 1m intervals giving up to 10 samples from shallow lakes (up to 10 m depth); greater intervals can be used for deeper lakes so that a total of 12 samples is obtained evenly spread over the water column.

Each sample consists of:

- One 0.5 L sample unfiltered (bottle A)
- One 0.5 L sample filtered (bottle B)
- One 0.4 L sample filtered and frozen (bottle C)

A single larger sample of 2 L (unfiltered) can be collected if filtration is to be carried out later in a land-based laboratory.

3.2 Sampling gear

- Peristaltic pump, pumping rate 100-300 ml/min, with silicone (or similar) pumping tubing; the pump could be powered by a battery (or by hand which will be slow).
- Teflon tubing, 0.5 cm ID, sufficiently long to reach the bottom of the lake (~ 20 m).
- The Teflon sampling tube should be weighed down with something heavy (rock in plastic bag?) suspended from the tube or a rope (nylon) to which the tube is also attached; the tube inlet should be about 50 cm above the weight. The bottom 30 cm of the tube should be unattached from the rope to enable the inlet to move away from the weight and the rope.
- Filtrations: 47 mm filter membranes, 0.4 or 0.45 μm pore size; Oxoid or Millipore polycarbonate are okay. Polypropylene, polycarbonate, polyethylene or Teflon filter holders

3.3 Sampling procedure

Sampling is most conveniently carried out using a peristaltic pump with an extended sampling tube lowered to the desired depth. The outflow of the pump is directed into sample bottles for sampling. The filtration is then conveniently carried during sampling by attaching an in-line filtration unit to the outflow of the pump. The sampling tube is flushed prior to sample collection by pumping water from the sampling depth for at least 5 mins. The water temperature should be monitored by holding a thermometer in the water flow. First a sample is collected for pH measurement. Then bottle A is rinsed three times with pumped water (~100 ml each time is sufficient) and is filled.

Filtration

An in-line filtration unit with a pre-cleaned filter is attached to the outflow of the pump and rinsed with lake water (about 100 mL or so); then the filtrate is directed into bottles B and C which are rinsed twice and then filled; bottle B can be filled fully but bottle C not fully to allow for expansion for freezing.

Alternative filtration in laboratory: peristaltic pump driven filtration is preferable with an in-line filtration unit. Alternatively pressure filtration can be used using an all polycarbonate (or other metal-clean plastic) filtration apparatus with the outflow

collected directly in a sample

bottle. It is also possible to pressurize a sample bottle with either pressurized air or nitrogen via a tube (silicone or similar) through the cap, and connect it via a second tube in the cap to an in-line filter holder. Vacuum is problematic as the filtrate should not arrive in a glass bottle.

Notes about touching sample bottles and pump tubes

Bottles are packed in plastic bags, and several bagged bottles are together in a second plastic bag. The outer bag is dirty, the inner one is clean and the bottles are very clean. The outer bag is opened and then transparent plastic gloves (clean ones) should be put on before the inner bag is opened. The bottle can be left partially in this bag whilst the cap is removed and placed inside the bag. The bottle is then rinsed and filled, then capped, the inner bag is closed and the bottle can be placed inside the partially open outer bag; the gloves are taken off and stored in a separate plastic bag for re-use, and the outer bottle bag is pulled up and closed.

The final part of the pump tube should be kept clean and should be touched only with gloves and can be rinsed with lake water. The entire pump tube should be stored in a plastic bag (large) when sampling is finished.

4. Radionuclides

Sampling will probably be carried out annually at three WP2 sites, Redo, Gossenköllesee and Neådalsvatn, by arrangement with Joan Grimalt (FBG). The object will be to determine particulate and dissolved ^{210}Pb concentrations in the water column. Using an INFILTREX II water sampler, c.500 L of lake water will be pumped through a 0.45 μm filter, and the filtrate passed through an in-line Axys Environmental Systems Ltd Radionuclide Type I exchange column. On completion, the filter should be carefully folded so as to ensure no loss of the retained particulates, packed and labelled, and sent with the exchange cartridge to Peter Appleby at University of Liverpool.

5. SCPs

Reference is the appropriate section of Neil Rose's SCP protocol, as also sited below:

At twice yearly intervals (end of summer, end of winter) at least 20 L of lake water should be filtered through a Whatman GF/C filter. The filter should be folded in half once (so that any filtered particles are enclosed) and stored flat in an individual plastic bag. If necessary, more than 1 filter can be used to filter the required sample volume. Sample bags should be clearly labelled 'lake water' with the site name, date and volume filtered and sent to Neil Rose at ECRC.

If possible a sample from the outflow could also be obtained in a similar way, and labelled 'outflow water'.

Volumes required may be subject to change after the initial analyses.

[If much larger volumes are needed, it may be appropriate to use the INFILTREX II water sampler (see Organic Micropollutants), by arrangement with Joan Grimalt.]

6. Organic Micropollutants

Sampling will probably be carried out annually at three WP2 sites, Redo, Gossenköllesee and Neådalsvatn, by arrangement with Joan Grimalt (FBG). The object will be to determine particulate and dissolved concentrations of organic micropollutants

in the water column. Using an, c.500 L of lake water will be pumped through a 0.45µm filter, and the filtrate passed through an in-line Axys Environmental Systems Ltd Trace Organics exchange column.

7. Suspended Sediments

[Measurements of suspended sediment concentrations (along with speciation studies) are essential for models of the behaviour of pollutants in the water column. They would need to be done by filtering large volumes (c.500L) using the INFILTREX II water sampler, by arrangement with Joan Grimalt. Practical details need to be sorted out this summer].