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European Mountain lake Ecosystems: Regionalisation, diaGnostics & socio-economic Evaluation

EMERGE

03

SURFACE WATER

SAMPLING AND ANALYSIS PROTOCOL

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Surface Water - Sampling and analysis protocol

1. Major ions and nutrients

The standard water samples are taken as surface grab samples from the outlet of the lake or in the lake centre where biological samples are taken.

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 strenght. 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.

3. Heavy Metals

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

You can collect samples either using a peristaltic pump, or using a special sample bottle (Niskin or Go-Flo) without metal containing parts on the inside, and preferably with Teflon coated parts on the outside. During the MOLAR project we had good results with both systems, but the sample bottles were most convenient.

- 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).
- Sample bottle: special Niskin bottle (1 or 1.5 L or larger), preferably modified for trace metal work by removing or Teflon-coating metal parts, and rubber parts from the inside of the bottle (silicone rings are okay). This bottle should be pre-cleaned using dilute acid (1 M HNO_3 or HCl), but an EDTA solution (1 mM) could be used too, followed by lots of rinsing with pure water.
- 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 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.

Sampling using a special sample bottle: pre-rinse the bottle several times with lake water, and operate it several times at the surface. Then lower the bottle to the desired depth, trigger it, bring it back up and empty it for cleaning. Now it can be used properly.

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.

It may be possible to pressurize the Niskin sample bottle and filter directly from this bottle into a sample bottle used for storage.

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 be carried 3-4 times per year annually at two WP3 sites, Redo and Ladove, by arrangement with Joan Grimalt (FBG). The object will be to determine particulate and dissolved concentrations of ^{210}Pb and ^{137}Cs in the water column. Using an INFILTREX II water sampler, c.200 L of lake water will be pumped through a 0.45μm filter, and the filtrate passed through an in-line Axys Environmental Systems Ltd Radionuclide Type I exchange column to measure ^{210}Pb and a Type II exchange column to measure ^{137}Cs. Measurements will be carried out at 3-4 depths. Careful records should be kept of the site, date, times, sampling depth volume sampled, and notes made of any problems encountered. On completion, the filters should be carefully folded so as to ensure no loss of the retained particulates, packed and labelled, and sent with the exchange cartridges and sampling records to Peter Appleby at University of Liverpool.

5. SCPs

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

Water column sampling:

- From WP3 - Redo and Ladove only.
- 4 or 10 depths; 4 times a year.
- Filter as much volume as possible (>100 litres if possible).
- Use GF/C filters. Use as small a diameter filter as possible. Use as few filters as possible.
- Along with the samples send a data sheet as follows:

Site Name	Site Code	Sample Date	Sample Depth	Filtered Volume
Round Loch	SCOT1	4/9/2000	1m	100 litres
Round Loch	SCOT1	4/9/2000	5m	105 litres

6. Organic Micropollutants

The object of this sampling 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.

There is something missing from this paragraph

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 will need to be done by filtering large volumes (c.200L) using the INFILTREX II water sampler, by arrangement with Joan Grimalt. Data will be available from the radionuclide (section 4) and organic micropollutant (section 5) measurements. Additional measurements using the pump without an exchange column should be carried out where this is thought necessary.