GUIDELINE FOR COLLECTION OF ENVIRONMENTAL SAMPLES FOR THE GREENLAND MINERAL RESOURCES ENVIRONMENTAL SAMPLE BANK

Technical Report from DCE – Danish Centre for Environment and Energy No. 239 2023

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Data sheet

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Abstract: This technical guideline includes technical instructions on sampling procedures for collection of environmental samples in relation to mineral resource projects in Greenland. The report also describes the registration of the samples. The aim of the technical guideline is to ensure that sampling is reproducible, comparable, and done according to international standards. This technical guideline replaces the instructions provided in the 'Guideline for collection of environmental samples to the Greenland mineral resources environmental sample bank Ver. 1.2, September 2022'.

Keywords:	Environmental monitoring, Collection of samples, Mineral resource projects, Greenland.
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Contents

Preface

This technical guideline includes instructions on sampling procedures for collection of environmental samples in relation to mineral resource projects in Greenland. The report also describes the registration of the samples and the procedure for handing over samples to DCE.

The aim of this technical guideline is to ensure that sampling is reproducible, comparable, and done according to international standards (e.g., Helcom 2017, OSPAR 2019). It replaces the instructions provided in the 'Guideline for collection of environmental samples to the Greenland mineral resources environmental sample bank Ver. 1.2, September 2022'.

Results from environmental monitoring of mining project in Greenland are registered in the Greenland mineral resources environmental sample bank hosted by Danish Centre for Environment and Energy (DCE) for the Greenland Authorities, The Environmental Agency for Mineral Resource Activities (EAMRA).

1 Introduction

The purpose of environmental monitoring of a mining project is to identify and quantify the environmental impact of the project. This technical guideline is prepared to ensure that Greenland samples are collected and sampled reproducible, comparable, and according to international standards.

The Section of Arctic Environment (DCE) at Aarhus University (AU) hosts the Greenland mineral resources environmental sample bank in Roskilde. This is done under a data agreement between the Greenland Authorities, The Environmental Agency for Mineral Resource Activities (EAMRA) and DCE. Samples have been collected for decades and are either stored frozen or as freeze-dried pulverized samples. The samples have been collected with the aim of elemental analyses for baseline sampling when preparing EIA or monitoring in relation to mineral resource exploration and extraction. A database hosted at the Section of Arctic Environment contains all sample information and in case the samples are analyzed at AU, the results of the analyses are also stored in the database. Please contact EAMRA and DCE for availability of specific data.

To ensure a high-quality monitoring program, the sampling should follow described standard procedures. Standardization is of high importance to ensure comparability between sample results spatially and temporally. This report includes technical instructions on sampling procedures for collection, pre-treatment, shipment, and storage of Arctic environmental samples. The report also describes the registration of the samples.

For monitoring purposes terrestrial and aquatic organisms, water, soil, sediments, and air are often sampled. This guideline includes procedures for sampling of:

- Lichens: crinkled snow lichen
- Mussels: blue mussels
- Seaweed: bladder wrack
- Fish: arctic char and sculpins
- Water: marine and freshwater
- Sediments: marine and freshwater sediments.

For other types of samples, e.g., soil, prawns, crabs, marine and terrestrial mammals, birds, eggs, etc., please contact the Section for Arctic Environment, DCE for instructions.

Contamination of samples must be avoided during collection, sample handling, and pre-treatment. As environmental samples are often collected along gradients around a contamination source, it is recommended (whenever possible) that samples are collected, pretreated etc. starting with the unpolluted samples first and following the gradient towards the pollution source to limit the risk of sample contamination.

It is important to note that if the samples are collected as part of a baseline study for a mining project in Greenland, and if the samples are not to be

analyzed by DCE, duplicate samples must be collected, and one set of samples must be sent to DCE to be stored in the sample bank for future reference.

To develop an environmental monitoring program please see 'Environmental monitoring at mine sites in Greenland, - A review of research and monitoring practices and their role in minimizing environmental impact' (Søndergaard et al., 2020).

2 Registration of samples

During collection, all samples should be registered by a unique ID number. DCE provides ID number books (each book holds 100 unique ID number pads). Therefore, the sampler should contact DCE at DCEMining@dce.au.dk to get hold of the necessary amount of ID books.

When sampling, all samples must get a specific ID number from the ID book. The ID pages must be filled out correctly and as complete as possible. For each ID number, there is an original page and a copy. When filled out, the original page must be stored together with the sample; the copy must stay in the ID book and will be archived at DCE. It is recommended that the original page is kept in a suitable plastic bag and packed with the sample. The ID number follows the sample, and analytical results will be archived by the ID number in the database. The ID number book need to be returned to DCE for archiving.

Besides the ID book, DCE provides a digital Microsoft Excel workbook with a number of sheets that also need to be filled out and sent to DCEMining@dce.au.dk. This includes the basic information recorded on the pages of the ID book, e.g., project name, collection date, sample type, species, and site/station, including GPS positions (latitude and longitude in decimal degrees and WGS84 datum). The excel workbook includes specific sheets for the different sample types, where more detailed information is entered. See Appendix A-E for an overview of the different sheets to be filled out.

Note that a correctly filled-in Excel file needs to be submitted to DCE before DCE can accept storage of the samples in the sample bank and EAMRA can approve the sampling as completed.

Figure 1. Orange ID number book and zip-lock plastic bags. The larger bag is used for the sample, while the smaller bag holds the sample ID number page. Photo by L. Bach.

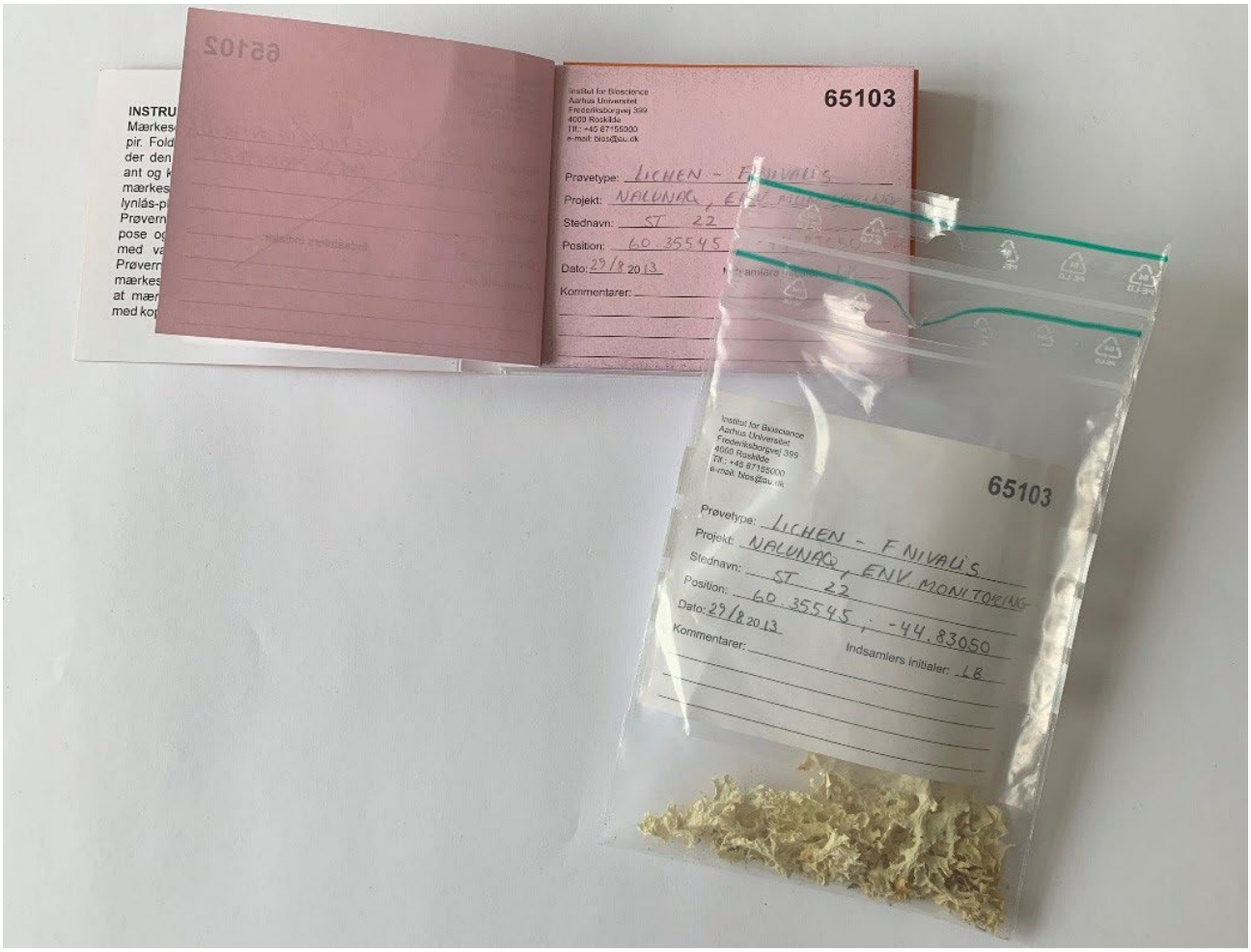

Figure 2. An example of a correctly packed sample, in this case a lichen sample. The smaller bag with the ID number sheet is placed inside the larger bag holding the sample to ensure the sample ID follows the sample. Photo by L. Bach.

3 Lichen sampling methodology

Lichens bioaccumulate atmospheric contaminants, such as metals, and are abundant in the Arctic (Søndergaard 2019; Søndergaard 2020). Their lack of roots, large surface area and long-life span enable lichens to effectively accumulate air contaminants and are therefore applied as a monitoring organism for air and dust pollution. The crinkled snow lichen, *Flavocetraria nivalis* is the preferred lichen species for monitoring purposes in Greenland.

Since the lifespan of *Flavocetraria nivalis* is several decades and due to a limited ability of lichens to excrete the bioaccumulated contaminants again, transplanted lichens have often been used as a supplement to, or instead of, resident lichens to assess the year-to-year variation in dust deposition (Søndergaard et al., 2013). Transplantation also makes it possible to monitor sites where no natural lichen occurs. Lichens to be transplanted are collected from uncontaminated reference sites and are typically placed at the monitoring sites for one year.

Figure 3. To assess dust deposition of contaminants from Greenland sites the lichen *Flavocetraria nivalis* is frequently used. Photo by L. Bach.

3.1 Sampling

At each sampling location (station), lichens are collected over an area of approximately 20m x 20m. Lichen samples should subsequently be sorted by hand using plastic tweezers and only fresh-looking yellowish parts of the lichens should be selected (either on-site or in a clean area in the camp or in the lab). Soil, debris and other organic parts than the lichen leaf-like branched structure should be discarded in the field.

A sample size of approximately a small hand of fine sorted material is sufficient. The sample should be stored in a zip-lock bag (polyethylene). Each sample is assigned a unique ID number. The ID number must clearly

follow the sample. The samples should be stored cold, and frozen at -18°C as soon as possible.

3.2 Sample registration

Data on lichen samples must be entered in the sheet named 'Basic' in the Microsoft Excel workbook provided by DCE (see appendix A). Besides filling out the standard fields in this sheet, be sure to make a note under comments in case the lichens were transplanted.

4 Mussel sampling methodology

Blue mussels are suspension feeders that filter large volumes of water through their gills (typically c. 3 liters per hour for an adult mussel) and feed mainly on phytoplankton (Famme et al., 1986). The contaminant accumulation in mussels is considered as an integrated measure of the concentration and bioavailability of both contaminants bound to particles and contaminants dissolved in the seawater (Beyer et al., 2017; Rainbow 1995).

In Greenland, the blue mussel (*Mytilus* sp.) occurs in shallow waters along most of the coasts of west Greenland, and as far north as ~70°N on the east coast. The species is therefore suitable for monitoring in near shore waters. Blue mussels in Greenland consist of the species *Mytilus edulis* and *Mytilus trossulus*. In general, *M. edulis* is distributed from the Disko Bay area and south, while *M. trossulus* is distributed from Disko Bay and northwards (Bach et al., 2019; Wenne et al., 2020). As the two species are indistinguishable by the naked eye, and the two species co-occur in some areas, the sampling should be with no distinction between the species.

If mussels are not natively present at the desired station for monitoring, they may be transplanted from an unpolluted area and then left at the desired station (Benedicto et al., 2011; Søndergaard et al., 2011) for e.g., one year before collection and analyses are conducted. The results will reflect last year's contamination impact in contrast to resident mussels that will reflect several years of contamination.

Figure 4. Blue mussel, *Mytilus* sp., bed at a rocky surface. Photo by L. Bach

4.1 Sampling

Mussels should be collected at the same time every year. In Greenland, sampling can most often be done from the coast during low tide hours (check the local tide schedule at DMI (https://www.dmi.dk/da/hav-og-is/tidevand/tidevandstabeller-for-groenland/). The samples at the different stations should be collected as close to the same depth and exposure (i.e., in terms of light and wave action) as possible to reduce variability in contaminant uptake.

At each station, 2 mussel samples each consisting of 20 specimens should be collected, representing two size groups. Preferred sampling size groups should cover the 3 to 6 cm size range (i.e., two of the following three size groups: 3.0-3.9; 4.0-4.9 and 5.0-5.9 cm shell length intervals). The sampled mussels should be cleaned from sediment, gravel, etc. attached to the exterior of the shell.

Mussels should be depurated prior to preservation. This is to facilitate the discharge of unassimilated particles in the mantle cavity or the gut that might significantly influence the metal concentration in the sample. This is especially important for mussels collected in water with high turbidity, on silt / clay bottoms.

***An important note for projects where baseline samples have consisted of un-depurated mussels**: For these projects, both un-depurated samples and depurated samples should be collected to enable comparison with previously collected samples. This 'duplicate' sampling is expected to cover a transition period of 3 years, where after only depurated mussel samples needs to be included in the sampling program.*

Seawater for depuration should be collected at each sampling station (10 liters per sample).

During transportation, the live mussels can be placed in buckets or bags and should preferably be kept moist if transported over long distances e.g., with a wet cloth or seaweed from the collection site. If the mussels have opened during transport and / or smell rotten, they must be discarded.

The shell length of each mussel in a sample is measured with a caliper for each individual and noted.

4.2 Depuration

The depuration must be started no later than 48 hours after collection.

For depuration, one mussel sample of 20 individuals should depurate in 10 L seawater. The sampled water (10 L) for depuration should be left for 6 hours for particles to settle in a 10 L bucket with a loose lid. Hereafter the water is decanted without any suspended particles into a 10 L depuration bucket. Afterwards the mussel sample is placed in a net (polyethylene) closed by a knot in the end. The mussels should be attached to the bucket in a way so the net hangs freely in the water above the bottom of the bucket. All buckets used should be polypropylene and approved for food storage.

The water should be aerated. This is important, as mussels will not depute in water low on oxygen. It is recommended to use a plug-in air pump; however, a battery driven air pump can be used in areas without electrical power. These pumps may be a little unstable, and it is therefore recommended to bring a spare.

The mussels should be left for depuration of particles for 20-24 hours.

Figure 5. An example of a setup for depuration of mussels. A mussel sample of 20 mussels is placed in a net in the water column of the bucket for 20-24 hours. Air is supplied using a battery powered air pump. Photo by L. Bach.

4.3 Dissection and storage

The mussels should be opened by cutting the muscles with a stainless-steel scalpel. The mussels should be inverted and allowed to drain on a clean towel or funnel for at least 5 minutes to drain excess water. Byssus threads should be removed, and the soft parts are then scraped out with a scalpel, taking care not to cut into the shells. The soft parts from 20 mussels of the same size class are pooled into a zip-lock bag (polyethylene). Usually, the shells are discarded. Each sample is assigned to a unique ID number. The ID number must clearly follow the sample. The samples should be stored cold, and frozen at -18°C as soon as possible.

4.4 Sample registration

Data on mussel samples must be entered in the two different sheets in the Microsoft Excel Workbook provided by DCE: 'Basic' (see appendix A) and 'Shellfish' (see appendix B). Besides filling out the standard fields in these sheets, be sure to make a note under comments in case mussels were transplanted. The habitat of the sampling site should be noted e.g., mussels collected on rocky shore, sandy beach, mud, etc.

In the Microsoft Excel Workbook provided by DCE, there is a legacy field recording sheet in Danish, which can be printed and used, when processing the mussel. However, for the data reporting to DCE, all information needs to be transferred to the sheets Basic and Shellfish.

5 Seaweed sampling methodology

In Greenland, three brown seaweed species dominate i.e., *Fucus vesiculosus* (Bladder wrack), *Fucus disticus* (Two-headed wrack or Common rock weed) and *Ascophyllum nodosum* (Knotted wrack). Sampling of *F. vesiculosus* should be preferred over *F. distichus*, which is preferred over *A. nodosum*. For identification of *F. vesiculosus* see figure 6, where the typical characters of *F. vesiculosus* with two parallel bladders are shown.

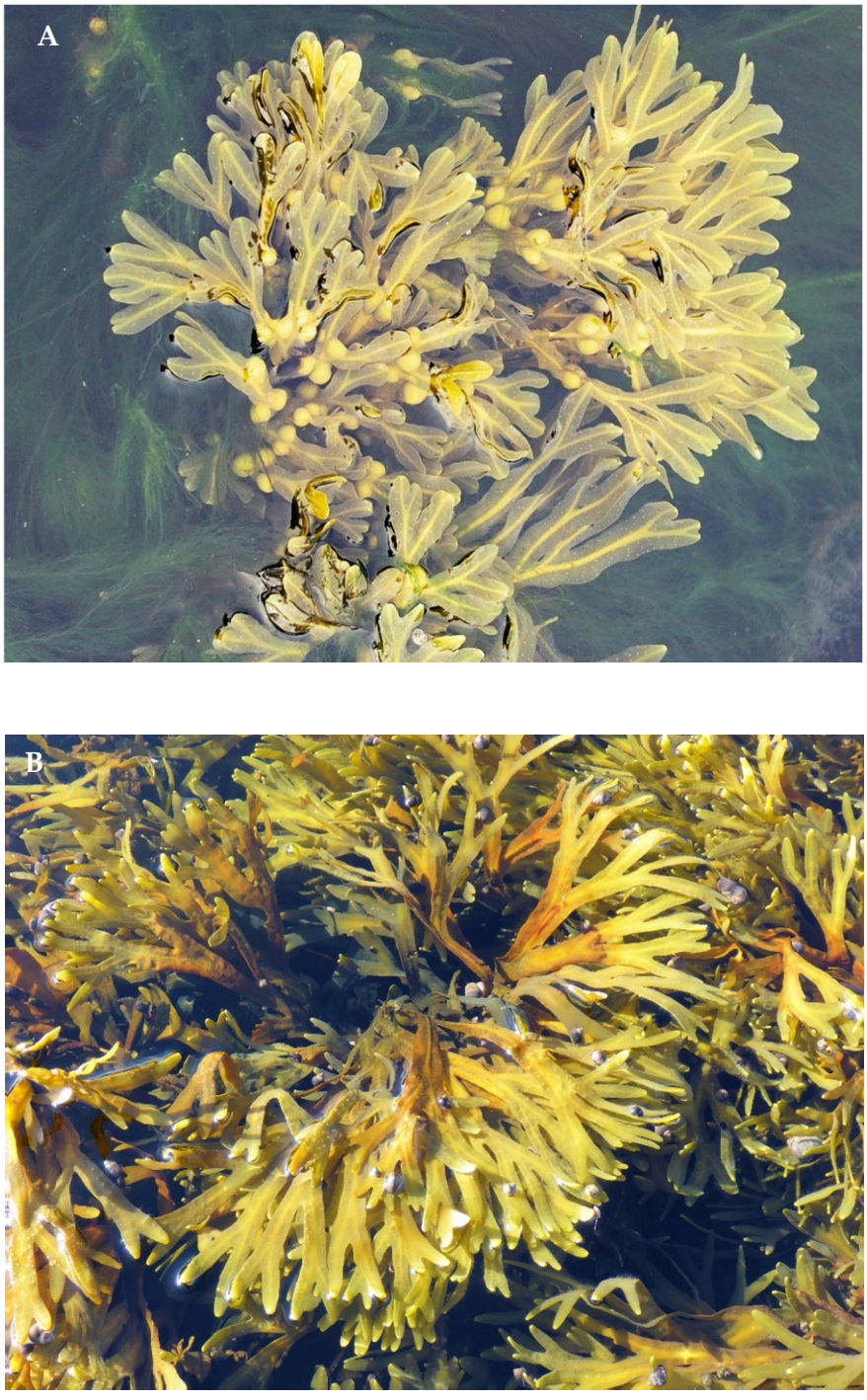

Figure 6. Photos of the three species, A) *F. vesiculosus*, B) *F. disticus* and C) *A. nodosum* for identification. Photos by O. Geertz-Hansen.

Growth tips of seaweed are used for monitoring purposes, as accumulation of metals in seaweed is regarded as a relative measure of the contaminant concentrations dissolved in the seawater (Rainbow, 1995). The growth tip contaminant concentrations reflect the accumulation in the present growing season (spring-autumn).

5.1 Sampling

Seaweed should be collected in August and September to be able to retrieve the new growth tips. Sampling of *Fucus vesiculosus* or *Fucus disticus* is most often possible from land during low tide.

Figure 7. The annual fresh growth tips of the seaweed are used as a proxy for the year-to-year variation in dissolved contaminants. Photo by O. Geertz-Hansen.

Check the local tide schedule at DMI (https: // www.dmi.dk / da / hav- og-is / tidevand / tidevandstabeller-for-groenland /). The samples at different stations should be collected as close to the same depth and exposure (i.e., in terms of light and wave action) as possible to reduce variability in contaminant uptake.

One seaweed sample should consist of approximately 100 grams of growth tips and comprise growth tips of 10–15 individual plants. Each sample is assigned a unique ID number. As a standard, two samples are collected per station (i.e., 2 ID numbers) with approximately 50 m between the two samples. At the site of collection, the collector should sample a good amount of seaweed.

After collection, the green-colored growth tips are cut off with stainless-steel (or aluminum) scissors until approximately 100 grams of growth tips are harvested (app. 2 handfuls).

The sample should be rinsed 3 times with deionized water (or alternatively tap water) to remove any associated biota such as snails and other organisms living on the seaweed. Subsequently, the sample should be placed in a zip-lock bag (polyethylene). The ID number follows the sample. The samples should be stored cold, and frozen at -18°C as soon as possible.

5.2 Sample registration

Data on seaweed samples must be entered in the sheet named 'Basic' in the Microsoft Excel workbook provided by DCE (see appendix A).

6 Fish sampling methodology

Fish species such as sculpin and Arctic char are often used in programs to monitor the marine and freshwater environment, respectively.

Sculpins (*Myoxocephalus* spp.) have been the preferred marine fish species used in the monitoring at Greenland mine sites because it is the most sedentary of the common fish species and abundant in both western and eastern Greenland. The shorthorn sculpin (*Myoxocephalus scorpius*) and fourhorn sculpin (*Myoxocephalus quadricornis*) are the most abundant species in Greenland.

Arctic char (*Salvelinus alpinus*) is a widespread fish in the Arctic and populates almost every lake and river in Greenland as either anadromous or landlocked populations. While landlocked fish stay in the freshwater system, the migrating fish will seek towards marine waters in May / June to feed, whereafter they will return to the freshwater systems in July/September. Here they will stay, usually within approximately 20 km of the system, where they overwinter (Muus et al., 1990). It can be difficult to identify whether a caught fish is a landlocked or migrating fish based on morphology. While landlocked fish are most often found in lakes, migrating fish are dominant in rivers. In general, the two forms can be distinguished by morphological divergences related to swimming performance and maneuverability. The landlocked Arctic chars are generally smaller, have a relatively larger head, smaller body, and longer caudal fin, as compared with migrating char (Damsgård 1991).

To apply fish as environmental monitoring organisms, analyses of the liver is often recommended, as it has an important role in contaminant storage, redistribution, detoxification, or transformation, and it is an important site of pathological effects induced by contaminants (Ewans et al., 1993). Muscle tissue is also often collected and is considered a proxy for a more recent uptake / accumulation of pollutants than the liver (Hansson et al., 2020). Muscle tissue is also preferred for studying transfer of pollutants in the food chain and thus the health aspects of human consumption.

6.1 Sampling

Fish, including arctic chars and sculpins, can most often be collected by angling, butmay also be caught by fine meshed nets on low water depths.

For ethical reasons, the fish must be killed with a blunt instrument or a knife through the head immediately after capture.

Five fish should be collected at each sampling site. Each fish is assigned a unique ID number.

Figure 8. Shorthorn sculpins, *Myoxocephalus scorpius*, are easily caught in most fjords in Greenland. As it is relatively sedentary, it is considered an indicator species for higher trophic level exposure. Photo by L. Bach.

The fish should preferably be processed right after collection. Alternatively, the fish should be stored cold until processing and maximum for 12 hours. When processing, the weight (to nearest gram) and total length (to nearest cm or 5 mm level) are measured, and the gender is determined. As the concentration of environmentally hazardous substances is different between males and females due to the transfer of some substances to roe during spawning, it is important that the sex is determined and noted. Males are usually smaller and have beautiful coloring, while the females usually are bigger and have faded colors. For Arctic chars, landlocked fish are preferred over migrating for monitoring purposes. If landlocked fish are not available, migrating fish can be used. In this case it should be noted clearly in the sampling spreadsheet.

Once the fish is open, the liver is sampled removed and rinsed in deionized water (or alternatively tap water). The liver is weighted (to nearest 0.1 g) and subsequently placed in a zip-lock bag (polyethylene). A muscle of approximately 4x2 cm is cut from the tail section. The muscle sample is placed in a zip-lock bag (polyethylene). Otoliths are collected using forceps after cutting the head from the dorsal side behind the eyes. The otoliths are stored in a zip-lock bag (polyethylene). All samples from the same fish (each marked with the same ID number) are placed in a third bag together with the ID number page. In case the fish are collected for analyses of organic contaminants, Rilsan™ nylon bags should be used. The samples should be stored cold, and frozen at -18°C as soon as possible.

Figure 9. Arctic chars are caught in rivers, often in river pools. Photo by L. Bach.

6.2 Sample registration

Biological variables as weight, length, sex, sexual maturity, stomach content, liver weight, liver condition i.e., parasites, color etc. that can have impact on the data assessment are measured and documented.

Data on fish samples must be entered in the two different sheets in the Microsoft Excel Workbook provided by DCE: 'Basic' (see appendix A) and 'Fish' (see appendix C).

In the Microsoft Excel Workbook provided by DCE, there is a legacy field recording sheet in Danish, which can be printed and used, when processing the fish. For the data reporting to DCE, all information needs to be transferred to the sheets Basic and Fish.

7 Water sampling methodology

Water samples are often collected as a part of monitoring programs, both from marine and freshwater environments such as streams, rivers, and lakes. Generally, both unfiltered and filtered (0.45 µm) samples are collected to provide information on total and dissolved concentrations of contaminants, respectively. In addition to water sampling for elemental analyses, measurement of pH, conductivity and total suspended solids are often conducted. Occasionally, the concentrations of suspended particulate-bound pollutants in the water are quantified and elements determined by analyzing the solid residue from a filtration process (Loring and Asmund, 1989; Søndergaard et al., 2011).

In recent years, passive chemical samplers (i.e., Diffusive Gradients in Thin films, DGT) have been used in addition to conventional techniques for measuring dissolved metals in both freshwater and seawater (Bach et al., 2013; Søndergaard et al., 2014). DGT samplers have the advantage that they provide a measure of the time-integrated and 'labile' metal concentrations during the deployment period as opposed to conventional water sampling that only provides a snapshot of the water chemistry. DGT samplers, however, only work for some metals depending on the type of device, and water quality criteria guidelines are typically only established for total metal concentrations in sea- and freshwater (MRA, 2015). Thus, for comparing data to water quality criteria guidelines, conventional water sampling is required. Consequently, DGT samplers should be regarded as a supplement to rather than a substitute for the conventional water sampling.

7.1 Sampling

Both an unfiltered and a filtered sample should be collected at the same station.

Seawater samples are typically collected at regular depth intervals (approximately every 5 or 10 m) from surface to bottom, using metal-free water samplers (like the Hydro-Bios standard or reversible water samplers / Niskine sampler) (Johansen et al., 2008). Freshwater samples are most often collected directly by hand in the sampling bottles / test tubes.

Regardless of the sampling technique, the sampler (Hydro-Bios standard or bottles) should be acid-cleaned prior to sampling.

The exact protocol for sampling of seawater and freshwater is very laboratory dependent as it depends on the specific analytical methods etc. Please consult the laboratory that will be analyzing the samples for instructions. For reference, DCE water sampling protocol is given in Appendix E.

Each water sample is assigned a unique ID number, i.e., a filtered and a non-filtered sample collected at the same station should have individual ID numbers. It is recommended to place the sample tube in a zip-lock bag (polyethylene). The ID number must follow the sample. The samples should ***not*** be stored frozen (except for nutrient analyses) and acid for preservation should generally ***not*** be added (but please consult the specific laboratory for this).

It is necessary to take into account that water samples need to be analyzed within 2 years after collection to be valid (Grasshoff et al., 1999).

Figure 10. Fresh water sampling. First a clean 1 L bottle is used to take a depth-integrated sample in the stream. Then a syringe is used to take subsamples of unfiltered and filtered water, the latter using disposable 0.45 µm syringe filters, into sample vials. Photo by O. Geertz-Hansen.

7.2 Sample registration

Data water samples must be entered in the sheet named 'Basic' in the Microsoft Excel workbook provided by DCE (see appendix A).

8 Sediment sampling methodology

Many environmentally harmful substances have a high affinity for particles to which they can adsorb / absorb and thereby settle out of the water phase. The sediment can re-suspend and gradually be transported by currents to a final sedimentation area. The sediment acts as a reservoir for a large group of environmentally harmful substances discharged into the marine or freshwater environment and is therefore a good proxy for monitoring pollution (temporal and / or geographical). In accumulation areas, both the current and previous pollution problems can be investigated, by sampling and analyzing segments of a sediment column. A sediment sample always represents a time period whose length depends on natural conditions such as sedimentation rate and bioturbation, i.e., animals digging in the sediment in the area.

Sediment samples are collected at fixed locations (stations) in the selected areas with a sampler capable of extracting a column of sediment. Information about the sediment such as color, layers, indication of bioturbations, appearance of the column, i.e., parameters that are important for the assessment of the result are noted. It is advised to use sediment core samplers. Using sediment core samplers, it can be ensured that the sampling method used does not mix the surface sediment (most recent deposited) with the underlying sediment (older sediments), as can occur by using different types of grabs (Xu et al., 2011). Sediment core samplers can be a HAPS-core sampler, a Kayak core sampler or others. Alternatively, samplers like the Ekman sampler for soft sediments, or a van Veen grab sampler for soft or medium-hard bottoms like sand, gravel, consolidated marl or clay, can be used if only the upper few centimeters of sediment are to be sampled (as a proxy of the recent deposition).

Figure 11. Sediment sampling of bottom surface sediment using an Ekman grab sampler. A slice of bottom surface sediment of 1-3 cm thickness can be obtained. Photos by J. Søndergaard.

8.1 Sampling

For both marine and freshwater sediment samples, the samples should as far as possible be collected in sedimentation areas. For marine areas, the sampling station should be > 50 m from point sources and the coast to avoid direct impact from land. For freshwater streams, rivers and lakes, the sediment samples should be taken in an area with low or no current, where transported particulate matter typically accumulates (sedimentation area). It is recommended to search the watercourse around the defined sampling station for the most suitable places to collect a sufficient amount of sediment.

For each station three sediment cores are collected to minimize the variability and impact of heterogeneous sediments. To assess the most recent contributions of contaminants, the upper section (or part of) of the sample (1-2 cm) should be carefully collected. It is important that only the fine sediment deposited is collected. This will be light and with a loose structure.

If the samples are taken in places with a low water depth (max 4 meters), kayak pipes can advantageously be used. In the case of a Kayak core sampler, the separation of the upper sediment is made after the surface sediment in the pipes has settled and the water is decanted. The decantation is preferably done by using a tube as this method allows for a more sensitive separation of water and sediment. Subsequently the upper 1-2 cm is carefully sliced off the core. If the sediment is very loose, the upper 1-2 cm can alternatively be transferred from the pipe with a tube or pipette.

If the samples are to be collected in rivers or streams with hard bedrock, the sampling can be conducted using a stainless-steel spoon. Three subsamples should be collected taking care only to scrape off the uppermost 1-2 cm. In freshwater systems, avoid getting coarse-grained gravel included in the samples.

Regardless of the sampling method, the three subsamples of sediment should be pooled, and the sample placed in a zip-lock bag (polyethylene). Sediment must be collected corresponding to a minimum of 75 g dry matter (approximately 200 g wet weight). The pooled sample is assigned one ID number. The ID number must follow the sample. The samples should be stored cold, and frozen at -18°C as soon as possible.

To acquire knowledge on deposition in time intervals, the entire sediment core should be divided fully into slices of 1-2 cm. In this case, each sediment core is assigned a unique ID number, thus all slices from the same core must have the same ID number. Each section of sediment (i.e., 0-2 cm, 2-4 cm, 4-6 cm etc.) is stored in individually zip-lock bags (polyethylene) marked with the ID number besides the depth of the slice. All slices are stored in one larger bag together with the ID number pad. Also write the ID number on the outside of the larger bag.

8.2 Sample registration

Sediment variables as water depth, distance from land, color, bioturbation, smell, grain size, etc. that can have impact on the data assessment are measured and documented.

Data on sediment samples must be entered in the two different sheets in the Microsoft Excel Workbook provided by DCE: 'Basic' (see appendix A) and 'Sediment' (see appendix C). Besides filling out the standard fields in these sheets, be sure to make a note under comments in case of signs of disrupted stratigraphy when visually assessed (signs of bioturbation). Also make a note if a photo of the core is captured.

9 Procedure for handling over samples to DCE

DCE stores environmental samples and data from mineral projects on behalf of the Environmental Agency for Mineral Resources Activities (EAMRA). The purpose of this chapter is to provide mining companies and their consultants with a clear, transparent description of DCE's procedure for reception, analyses and storage of environmental samples and data in DCE's sample bank and in the Mineral Resources Environmental Database (MRED).

Procedures are given for three categories of samples:

- The mining companies' baseline samples.
- Environmental monitoring samples collected by the mining companies during the mining phase as part of their self-control program.
- Environmental monitoring samples collected during the mining phase and after closure as part of the Environmental Authorities' control program performed by DCE/GINR.

9.1 Procedures for samples collected by mining company as part of the baseline program

Procedure for reception, analyses and storage of **baseline samples and data for mining companies** as part of their baseline studies and Environmental Impact Assessment (EIA) work:

1) Prior to sampling, the mining company contacts DCE at DCEMining@dce.au.dk to receive a manual for sampling, a Microsoft Excel workbook for recording sample information, and sample ID number books.

2) After sampling, the mining company delivers one set of duplicate samples to DCE for storage (after agreement) and keeps the other duplicate samples for their own storage and subsequent analyses. Duplicate samples can e.g., be fish liver samples split into two equally sized parts or replicate samples of seaweed, mussels and lichens. If duplicate samples are not available and only one sample exists, DCE stores the samples.

3) Along with the samples, the mining company sends the preformatted Microsoft Excel workbook with sample information and the used ID number book. DCE can only accept storage of the samples in the sample bank after a correctly filled out Excel file has been received.

4) DCE achieves the ID number book, performs quality control of the sample information in the preformatted Microsoft Excel workbook, and formats and adds the data to the MRED database. Before storage, DCE also performs a check that the received samples match what is listed in the Excel file. Upon completion of these task, DCE reports back to the company and EAMRA on reception of data and samples, shortly listing what been received and highlighting insufficiencies if such were encountered.

5) *For water samples only*: DCE notifies EAMRA that the samples have been

received, are stored in the sample bank and can be analyzed within a period after 2 years after collection in case EAMRA wishes to do so as a control. Due to the limited chemical stability of water samples, analyses should be performed as soon as possible and not later than 2 years after sampling given that the samples have been properly acid preserved (Grasshoff et al., 1999).

Subsequently, for freshwater samples, 1 ml of clean nitric acid (Suprapure or Ultrapure quality) per liter of sample is added to preserve the samples. For seawater samples, 2 ml per liter of sample is added. After 2 years of storage, and if nothing else is agreed, DCE will contact EAMRA to discuss whether the samples can be discarded.

6) *For all other samples than water samples*: DCE stores the samples in the sample bank until further instructions from EAMRA. In case EAMRA finds it relevant to analyze the samples, or just a selection of the samples, for control, this can be done at any time.

7) The company decides if and when chemical analyses shall be made, often as a step in the preparation of an EIA report. It can also be upon request from EAMRA. If the company moves on with chemical analyses of the samples, the laboratory performing the analyses should be accredited to do the chemical analyses and the lab's detection limits for environmentally important elements should comply with the Danish Environmental Protection Agency's current requirements for environmental measurements (in Danish: "Bekendtgørelse om kvalitetskrav til miljømålinger", available at www.retsinformation.dk). For samples already stored by DCE, and if duplicate samples are not available, DCE can send a subsample to the mining company for a fee covering DCE's expenses for sample preparation. For seaweed and mussel samples, the sample preparation will involve freeze-drying and homogenizing of the samples to provide a representative subsample.

8) The company sends analysis results as an excel sheet to DCE following a DCE template.

9) DCE checks that the excel sheet has been filled out correctly and adds the analysis results to the MRED database. DCE performs a screening of the results to evaluate the quality of the data (DCE can of course not be held responsible for the quality of other lab's data). In case concerns arise about the analytic results or lacking information, DCE notifies EAMRA. If EAMRA wants to check the quality of the lab's results, DCE can analyze samples for control.

9.2 Procedures for samples collected by mining company as part of self-control program

Procedure for reception, analyses and storage of **environmental monitoring samples and data** collected during the mining phase as part of the **Mining Companies' Self-control Program**.

Unless specifically requested by EAMRA, samples from the Mining Companies' Self-control Program will not be included in the DCE's sample bank and database. Thus, samples from Mining Companies' Self-control

Program shall not be sent to the DCE. Analytical results of samples from the Mining Companies' Self-control Program shall be sent to EAMRA and will be forwarded to DCE for filing, but the data will not be included in the MRED database.

9.3 Procedures for samples collected by environmental authority as part of monitoring program

Procedure for reception, analyses and storage of samples and data for **environmental monitoring samples** and data collected during the mining phase and after closure as part of the **Environmental Authorities' Control Program performed by DCE/GINR**.

1) Samples collected by or by representatives for DCE/GINR are delivered to the DCE after agreement, as are the preformatted Microsoft Excel workbook with sample information and the used ID number book.
2) DCE achieves the ID number book, performs quality control of the sample information in the preformatted Microsoft Excel workbook, and formats and adds the data to the MRED database. Before storage, DCE also performs a check that the received samples matches what is listed in the Excel file.
3) *For water samples only*: DCE stores the samples in the sample bank until chemical analyses. Prior to storage, for freshwater samples, 1 ml of clean nitric acid (Suprapure or Ultrapure quality) per liter of sample is added to preserve the samples. For seawater samples, 2 ml per liter of sample is added. Since water samples have a limited chemical stability, analyses should be performed as soon as possible and not later than 2 years after sampling given that the samples have been properly acid preserved (Grasshoff et al., 1999).
4) *For all other samples than water samples*: DCE stores the samples in the sample bank.
5) EAMRA decides if and when the chemical analyses shall be made.
6) The DCE lab performs the chemical analyses.
7) DCE adds the chemical analysis results to the MRED database.
8) DCE prepares an analyses report to EAMRA, including an interpretation of the results as per agreement with EAMRA.

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Appendix A – Basic registration for all sample types

For all samples collected, regardless of type, information from the ID number book must be entered in the sheet named "Basic" in the Microsoft Excel Workbook provided by DCE. The sheet contains help text explaining to how to fill out the different fields, and in some cases field values are constrained to certain predefined codes. All fields need to be filled out. In case of uncertainty on the registration, please contact DCE.

Hypothetical example of data recorded in the Basic table

ID no	License no	Mining company	Sampling company	Project	Project part	Sample type	Collection date	Station	LatDecDeg	LonDecDeg	Collector	Collection method	Comments
15126	MEL 1962-15	Black Angel Mining	Green water	Maarmorilik	1992 monitoring	Blue mussel	1992091	T12	71.13766667	-51.24033333	John Bly	Skin diving	40-49 mm
15127	MEL 1962-15	Black Angel Mining	Green water	Maarmorilik	1992 monitoring	Crinkled snow lichen	1992091	T12	71.13766667	-51.24033333	John Bly	By hand	
15128	MEL 1962-15	Black Angel Mining	Green water	Maarmorilik	1992 monitoring	Bladder wrack	1992091	T12	71.13766667	-51.24033333	John Bly	Skin diving	
15129	MEL 1962-15	Black Angel Mining	Green water	Maarmorilik	1992 monitoring	Shorthorn sculpin	1992091	T12Ø	71.13766667	-51.24033333	John Bly	Angling	
15129	MEL 1962-15	Black Angel Mining	Green water	Maarmorilik	1992 monitoring	Sediment	1992091	T5	71.26185	-51.326687	John Bly	Kayak corer	

For certain sample types (blue mussels, fish and sediments) additional Excel sheets also need to be filled out (see appendix B, C and D).

Appendix B – Additional registration for shellfish samples

For mussel samples, not only the "Basic" sheet needs to be filled out, but also a sheet called "Shellfish", which contains special information pertaining only to this sample type. The sheet contains help text explaining to how to fill out the different fields, and in some cases field values are constrained to certain predefined codes. All fields need to be filled out. In case of uncertainty on the registration, please contact DCE.

Hypothetical example of data recorded in the Shellfish table

ID no	No of individuals	Shell length min (mm)	Shell length max (mm)	Shell length average (mm)	sample wet weight (g)	Depurated	Comments
25317	20	40	49	43.5	82	Yes	
25318	20	50	59	54.2	93	Yes	
25319	20	40	49	44.1	88	No	
25320	20	50	59	55.2	97	No	

Appendix C – Additional registration for fish samples

For fish samples (sculpins and char), not only the "Basic" sheet needs to be filled out, but also a sheet called "Fish", which contains special information pertaining only to this sample type. The sheet contains help text explaining to how to fill out the different fields, and in some cases field values are constrained to certain predefined codes. All fields need to be filled out. In case of uncertainty on the registration, please contact DCE.

Hypothetical example of data recorded in the Fish table

ID no	Sex	Total length (cm)	Weight (g)	Liver sampled	Muscle sampled	Otoliths sampled	Liver weight (g)	Stomach content	Char pop	Comments
34073	Female	34.5	581	Yes	Yes	No	25.43	Crabs		From polluted area
34074	Female	23	204	Yes	Yes	Yes	13.15	Empty		
34075	Male	26	258	Yes	Yes	Yes	18.33			
34076	Female	52	2102	Yes	Yes	Yes	21.51	Capelin	Anadromous	
34077	Male	22	95.4	Yes	No	No	1.72	Empty	Landlocked	

Appendix D – Additional registration for sediment samples

For sediment samples, not only the "Basic" sheet needs to be filled out, but also a sheet called "Sediment", which contains special information pertaining only to this sample type. The sheet contains help text explaining to how to fill out the different fields, and in some cases field values are constrained to certain predefined codes. All fields need to be filled out. In case of uncertainty on the registration, please contact DCE.

Hypothetical example of data recorded in the Sediment table

ID no	Water depth (m)	Slice size (cm)	Number of slices	Core height (cm)	Sieved	Mesh size (mm)	Comments
40011	10	5	2	10	Yes	1	Polluted area, photo is captured
40114	65	2	20	40	No		Shells, photo is captured
40115	70	2	10	20	No		Corer hit rock at bottom

Appendix E – Water sampling

Instruction for sampling and filtering of water for trace metal analyses in DCE's accredited laboratory.

Equipment:

- Laboratory gloves
- A large clean (acid-washed) polyethylene sampling bottle (typically 1-2 l)
- Disposable polypropylene / polyethylene syringes (without rubber O-ring, which is prone to Zn carry-over)
- Syringe filters (nylon or PES, 0.45 µm pore size)
- Sample vials / bottles. For trace metal analyses (excl. Hg) in freshwater: typically, 15 ml polypropylene ICP-MS vials. For trace metal analyses (excl. Hg) in seawater: typically, 100 ml or larger polyethylene bottles. For Hg analyses in fresh- and seawater: typically, 100 ml or larger glass or PTFE bottles. It is usually an advantage to mark the vials / bottles with sample ID using a water-resistant marker beforehand.
- Polyethylene bags for sample vials / bottles.
- Possibly ID labels, GPS, water resistant marker or other writing tools for marking the samples.
- Possibly a water sampler (like the Ruttner-type sampler) if sampling of seawater along with cord and messenger load.

Method:

1) Wear laboratory gloves during sampling and be cautious to avoid touching surfaces that get in contact with the sample water during sampling.

2) Rinse the large sampling bottle 3 times with water similar to the water that will be sampled.

   Depending on where the sample is taken or if it is fresh- or seawater, the sample can be taken 'by hand' using the large sampling bottle e.g., in a freshwater stream or using a dedicated water sampler (like the Ruttner type sampler) for depth-specific sampling in the sea.

   In a freshwater stream it is usually preferred to obtain a depth-integrated sample by moving the sampling bottle up and down though the water column until it is filled. Avoid surface water which can contain debris like insects, leaves etc. Also avoid kicking up and suspending sediment from the bottom of the stream, which can contribute to an elevated particle concentration in the sample.

   In the sea, depth-specific sampling is usually conducted using various water samplers (like the Ruttner type) equipped with a messenger load released when the sampler reaches the desired depth. It is important that the water container of the sampler is made entirely of plastic (i.e., not metal parts in contact with the sample) if the sample is to be analyzed for trace metals. Once the sampler is retrieved to the surface, transfer the water to

the large sampling bottle.

3) Pack out a syringe and rinse the syringe 3 times with the water from the sampling bottle (i.e., fill and empty the syringe 3 times and discard the water).

4) Fill the syringe and take an optional unfiltered sample by transferring the content to a vial / bottle (recommended).

5) Pack out a filter, fill the syringe, put the filter on and rinse the filter by pressing the volume of water in the syringe though the filter once and discard the water.

6) Fill the syringe, put the filter on again and press the water into the sample vial / bottle to take a filtered sample. It is often an advantage to press the filter against the edge of the vial / bottle when the water is pressed though it to avoid the filter detaching from the syringe under pressure. If the water contains high concentrations of particles, it may be necessary to use more than one filter per sample. In that case, remember to also rinse these filters before use. Fill the sample bottle entirely.

7) It is usually preferred to take duplicate samples such as two unfiltered and two filtered samples (using two separate filters) at each location.

8) Make sure the samples are clearly marked with sample ID. In addition, it is usually preferred to put the sample vials / bottles in a separate plastic bag with the sample ID written on the bag also.

9) Store the samples cool if possible until they reach the lab. DO NOT FREEZE THE SAMPLES as the lid may break and metals may precipitate during the freezing process. Once the samples are received in the lab, they will be preserved with clean acid prior to analysis.

GUIDELINE FOR COLLECTION OF ENVIRONMENTAL SAMPLES TO THE GREENLAND MINERAL RESOURCES ENVIRONMENTAL SAMPLE BANK

This guideline includes technical instructions on sampling procedures for collection of environmental samples in relation to mineral resource projects in Greenland. The report also describes how record and report data on the samples. The aim of the guideline is to ensure that sampling is reproducible, comparable, and done according to international standards. It replaces the instructions provided in the 'Guideline for collection of environmental samples to the Greenland mineral resources environmental sample bank Ver. 1.2, September 2022.

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