3. Preparation of Samples

3.1 Sampling, preparation and storage

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Representative sampling is necessary for especially heterogeneous samples such as soils and sediments. This is feasible by several punctures for soil samples or by collecting several sediment cores and the subsequent mixing of several samples. Samples can be filled in plastic bags or transported directly in the sediment tube. If further analyses are necessary, e.g. concentrations of plant protection products, transport bags/boxes have to be checked for possibility of (de)sorption of analytes and replaced if necessary. A very inert material is Teflon (polytetra fluor ethylen). If heavy metal concentrations have to be determined parallelly each contact with metal-containing material have to be avoided (e.g. sampler, sieves, spatula, …).

3.1.1 Solid sample material (with low water content)

After sampling, material has to be chopped coarsely and dried in flat bowls. For very loamy or clayey soils as well as for peat chopping is essentially because such material will otherwise cake to hard lumps during drying, making chopping nearly impossible.
If other analyses are necessary, e.g. molecular biological analyses, samples have to be prepared according to the most sensitive analysis method. This may be the immediate preparation/analysis of fresh tissues but also freezing. Soil samples are sieved < 2 mm after drying and if necessary, sub samples are grinded. After drying, sediments are grinded as well and optionally ashed depending on digestion method (e.g. persulfate digestion). In this case the loss of ignition has to be determined.

3.1.2 Water-rich sample material

Phosphorus compounds in liquid samples can react from bond phosphorus to phosphate (desorption, phosphatases) or free phosphate can be bonded (absorbed in cells, adsorption). To avoid such processes or at least reduce them samples have to be transported refrigerated and be stored in a refrigerator at 4 °C (maximum a few days). The best storage is freezing (-20 °C). In samples with high water content the percentage of dry matter or water content have to be determined. After lyophilisation or drying

samples are milled. Elemental concentrations are determined after ashing at 550 °C.
The loss of ignition has to be determined for ashed samples to calculate the P concentrations in dry matter or sample volume from those in ash. Water samples are not dried but prepared wet chemically. Particulate organic matter (POM) can be concentrated on glass fibre filter. However, it has to be considered that the (low) P concentrations in these filters increase the blanks significantly.

3.1.3 Biomass: algae, plants and animal tissue

In comparison to higher plant algae have less supporting tissues. Therefore, it is easy to chop and homogenise algae biomass. Due to the lack of differentiation of thalli it is not necessary and not possible to analyse different parts of the organism.
The different composition of plant tissues (supporting tissue or storage tissue) result in different analyses of the different plant parts according to the scientific question. Very coarse or wet plant material such as potato tubers have to be chopped (the peel optional separately) before drying (by air or in drying oven) or lyophilisation. After drying plant material has to be grinded stepwise (first coarse, then finer mills, especially for woody material) and/or ashed.
Animals such as mussels or fishes have to be transported refrigerated and must be frozen quickly. Under certain circumstances, separation in meat and bones for fishes or meat and shells for mussels can be necessary. Bones and shells can be dried in a drying oven and be milled subsequently. Meat of fishes and mussels produces a lot of odour during ignition in moist status and a lot of soot by fat burning. It can be tried to decrease the problems by previous freezing and lyophilisation. Alternatively, a new method for ashing in a microwave system is available (Phönix, Fa. CEM). The exceptionally high concentrations of P in bones means that small amount of ash have to be weigh in (poor reproducibility) or measurement solutions have to be strongly diluted (poor reproducibility and dilution secondary errors). For this reason, it is interesting to work on digestions of dry matter and optimize the digestion method as well as quantifying yield.

Reference

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