3. Preparation of Samples

3.3 Ashing and loss of ignition

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Since concentrations of soil organic matter (SOM) in soils and some muds are < 15 %, ashing is not necessary and sometimes counterproductive. The conversion of minerals such as goethite to hematite can result in stronger binding of P (covalent, Cornell & Schwertmann 2006). Crystal water from clay minerals is removed and particles can shrink and include elements. Samples with > 15 % SOM (Blume et al. 2010) can be ashed, but samples with > 30 % (peat) should be ashed. If ashing is not possible, such organic rich samples have to be digested very strongly (microwave, H_2O_2 plus HNO_3). Animal material (except bones) should be ashed or if digested by microwave they should be lyophilized and milled very fine.

High Fe concentrations in soils and sediments cause a red sample colour during ashing, since between 500 and 600 °C yellow-brown ferrihydrites or goethite is converted to red hematite (Derie et al. 1976). Such red colouring can complicate neutralisation after persulfate digestion, since the indicator for the pH value is yellow. At high Fe concentrations in the extract, Fe-flakes can appear during neutralisation with NaOH or ammonia. These Fe-flakes can absorb parts of the P and remove it from photometric detection or increase turbidity during photometric detection (if not completely dissolved before).

During ashing of animal material, such as fish or mussels, huge amounts of soot develop in muffle furnace. This is caused by the high percentages of fat in the biomass. Due to the differences in composition of matrix, mussels have to be separated into mollusc flesh and shells. High carbonate concentrations can disturb the digestion of samples (cf. chapter 2.2). Phosphorus concentrations in vertebrates are often only presented for (human) used tissues. If balances are needed, P concentrations of bones are necessary as well. In the ScienceCampus Phosphorus Research there is only little experience with pre-treatment and extraction of animal products such as fish.

Before ashing, samples have to be dried (or lyophilised) until constant weight. Longer stored material (weeks to months) have to be dried again before weigh-in. For ashing dry matter weigh and ash weigh have to be

noticed to determine the loss of ignition. Only by this value elemental concentrations can be calculated for dry matter.

Protocol

- Determine empty mass of crucibles
- Weigh in sediment or dry biomass (estimation of amount in table 3.3-1)
- Dry longer stored dry matter for some hours in desiccator before weigh-in
- Note following masses: crucible empty, crucible with dry matter and crucible with ash
- Put crucible in the muffle furnace for 4 hours at 550 °C,
- Attention: very long time for cooling down in oven > 12 h, transfer to desiccator only with crucible pliers!!!
- Cooling down crucible in desiccator and weigh
- Before calculation of loss of ignition, calculate all masses without crucible mass!
- Minimum mass of ashes is 50 mg, to avoid complications by contaminations
- It is safer to calculate stepwise (see example), because neither crucible masses nor conversion of ash (g absolute), loss of ignition (% dry matter) can easily be excluded or reformulated.

Table 3.3-1 Common weight for dry matter for ashing of material

Drying		
	Weigh-in of dry matter (ca. g)	**Loss of ignition (%)**
Soils	less common	>90
Sediment	1	20-90
Algae	0,5	10-40
Plants	0,5-1	
Animal tissue		

Equation 3.3-1 Calculation of residue of ignition in % dry matter

$$RoI = \frac{A}{DM} \times 100\,\%$$

A Ash (g or mg)
DM Dry Matter (g or mg)
RoI Residue of Ignition (%)

Equation 3.3-2 Calculation of loss of ignition in % dry matter

$$LoI = 100\ \% - RoI$$

LoI Loss of Ignition (%)

$$RoI = \frac{Cr_{out}\text{-}Cr_{empty}}{Cr_{in}\text{-}Cr_{empty}} = \frac{27.198\text{-}26.087}{27.587\text{-}26.087} \times 100\ \%$$

$$RoI = \frac{1.111}{1.5} \times 100\ \%$$

Cr_{in} Crucible weigh-in with dry matter (g)
Cr_{out} Crucible weight out after ashing (g)
Cr_{empty} Crucible empty mass (g)

$$LoI = 100\ \% - 74\ \% = 26\ \%$$

The loss of ignition is the organic matter of samples which is lost by ignition (ashing) (elements C, H, O and N).

References

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