

Mineralisation of plant material for determination of metal content




Principle

The method for determination of total plant U and macro and micro elements described here is a combustion and treatment with HCl. This method requires a muffle furnace but is safer and less expensive than use of an aggressive mixture of acids.

Dried plant sample is calcined and the ashes are dissolved in concentrated HCl. This is then diluted to a given volume, filtered and ready for analysis.

Donohue, S.J., Friedericks, J.B., 1984. Laboratory Procedures. Soil Testing and Plant Analysis Laboratory, Virginia Technical Publication, pp. 452-881.

Equipment and Reagents

Machine/Product	Reference (Company, Type, ...)
Programmable muffle furnace	 Carbolite Eurotherm 2416CC
Heat-resistant glass vials	
Heat-resistant marker (GP-X)	
Plastic syringes (disposable)	
Syringe filters (acid compatible)	Acrodisc® HT Tuffryn® 0.45 µm
Drying oven	Memmert
Hydrochloric Acid concentrated HCl 37 %; 1,19 g.ml ⁻¹ ; 36.46 g.mol ⁻¹ = 12.08 mol.l ⁻¹	  VWR 20252.290

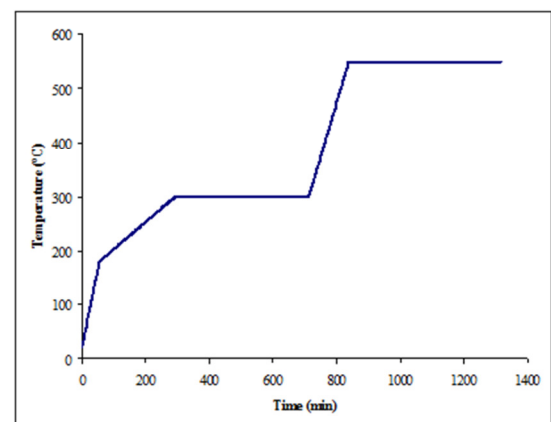
Procedure

Manipulations

- Oven-dry the plant sample (70 °C, one week)
- Weigh approximately 4 g (+ 0.001 g) oven dry plant sample into a tared 20 ml glass vial (or into another suitable heat-resistant recipient) (= A gram net plant sample). Smaller samples should be weighed on an analytical balance with a precision of 0.1 mg.
- Calcine the sample by running it through the LOI-program in a muffle furnace.

LOI – program

- heat up the oven at a rate of 3 °C.min⁻¹ for about one hour up to 180 °C
- continue heating at a rate of 0.5 °C.min⁻¹ for about four hours up to 300 °C
- keep the temperature on 300 °C for 7 hours
- heat up to 550 °C at a rate of 2 °C.min⁻¹ for about two hours
- keep the temperature at 550 °C overnight.
- It is best to let cool off the recipients outside the desiccator for a few minutes to prevent the desiccator to heat up needlessly.
- Allow the samples to cool down to room temperature.



Dissolving the ashed material

- Dissolve the ash by cautious addition of 1.5 ml HCl_{conc} (36-37 %) and let stand for 30 minutes. Warning: Concentrated HCl can cause severe burns and emits corrosive fumes. Gloves and eye protection are advised when using concentrated acids. Work under a fume hood.
- In case of very small samples (below 500 mg) the dissolution is carried out with 1 ml 1 M HCl, heat on a sand bath, (#5) wait for the dissolution to be complete (in about 30 min there should not be any black particles left), then add 9 ml H₂O. If the dissolution is not complete, bring to dryness on the sand bath and repeat the calcination step and the dissolution procedure.
- Cautiously add 15 ml demineralized water.
- Prepare blanks by diluting 1.5 g HCl_{conc} in 15 ml demineralized water.
- Weigh the vial with the solution. (= B gram net solution)
- Filter blanks and sample through syringe filters.
- Macro and micro elements can be determined by AAS, ICP-MS or ICP-AES. Phosphorus can be determined using the molybdate-blue spectrophotometric procedure.

Calculation

The element analyzed ($\mu\text{g/g}$ dry sample) is obtained by:

$$\text{concentration element } (\mu\text{g} / \text{g}) = \frac{(C - \text{blank}) \times B}{1000 \times A}$$

Where:

A = gram net oven dried plant sample

B = gram net solution + dissolved ash

C = element concentration in filtrate in $\mu\text{g.l}^{-1}$

Remarks

- The quantities sample and acid used in this method can be varied to fit specific needs of analysis, e.g. to use more sample when available or concentrate the filtrate for micro-elements analysis.
- Sometimes it is required to heat the samples on a sand bath to complete the dissolution of the ashed sample. To keep uniformity in the sample composition it's important to keep track of blanks and the evaporation loss of acid.