

### Mode opératoire

Réf :

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## Mineralization using the HotBlock

### 1. Object and application domain

Mineralization samples (MineEnglish) with Hotblock for atomic analysis.

**2. Diffusion list**: BPMP, others

### 3. Health and Safety

Wear labcoat, gloves and glasses (nitric acid concentrated manipulation ). All steps under the fume hood.

### 4. Method principle

It is an acidic hydrolysis (mix of nitric acid and  $H_2O_2$ ) at high temperature (85°C) in a hotblock (maximum 96 samples).

### 5. Required material

Precision balance (room 236 MeMo lab), TPE tubes for the Hotblock (supplied by SAME).

### 6. Chemical and Biological Reagent

Nitric acid 65% (acid cabinet Memo room 237) and hydrogen peroxide ( $H_2O_2$  at 4°C Memo's frige room 238), supplied by SAME.

### 7. Method complexity

Hazardous products manipulation: nitric acid and H<sub>2</sub>O<sub>2</sub>, hotblock.

#### 8. Procedure

Vegetal material: between 5mg (seeds) and 20mg (roots, leaves) of dried material.

For an optimal mineralization, gently reduce the tissues into a powder (mortar & pestle, manually or with a grinder, avoid metal beads or tools...).

Weight TPE tubes with their caps.

Weight the dried samples in the TPE tubes.

**I Préparation** : All steps under the fume hood, the day before the acidic digestion.

Add 750µl of Nitric Acid 65%, gently homogenize.

Add 250µl of H<sub>2</sub>O<sub>2</sub> 30%.

Gently homogenize.

#### Degas OVERNIGHT at room temperature, under the fume hood.

Wait until degas is complete between 8 to 16 hours, no more visible bubles when the tubes are gently homogenized.

The caps can be disposed on the tubes but not screwed and degas overnight.

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#### II The next day, Close the device :

Screw the cap to close the tube, not too tight; the cap must be unscrewed a quarter of a turn.

### **III Starting-up:**

- 1. Transfer tubes on the rack in the Hotblock.
- 2. Switch the hotblock on (button on the right side of the machine).

The temperature appears on the left side of the machine.

Be carefull: the tubes must be in the hotblock when the ramp is starting. It takes about 35 minutes to reach 85°C (SoAK) which last **24 hours**.

IV End of the Run: The Hotblock is programmed to last 24h, if mineralization is complete after 8h (depend on sample type) switch the hotblock of.

- 1. Remove **gently** the rack with tubes from the block, put them under the fume hood.
- 2. Once cool, screw the caps.
- 3. Tap the tubes to fall down the drops in the cap. If necessary, vortex or shake vigorousely.
- 4. *If the liquids are transparent* (can be yellowish), the samples are completely digested (no particles remaining). Samples must be diluted with 4 ml MilliQ H<sub>2</sub>O.
- 5. if particles remaining: start again the RUN.
- 6. Weight the tubes containing mineralized samples and caps : measure final volume (5 g ⇔ 5ml).
- 7. For MP-AES Analysis, transfer samples in the in the convenient tubes for the autosampler (Agilent PPP tubes supplied by SAME).

The samples can be stored in the cold chamber.

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