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	Mineralization of Recalcitrant Samples by acidic digestion using the SPEEDWAVE	

1. Object and application domain

Mineralization for recalcitrant samples (MER) with Speedwave 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 H₂O₂) at high temperature (180°C) in a micro-wave « Speedwave » (maximum 24 samples).

5. Required material

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

6. Chemical and Biological Reagent

Nitric acid 65% (acid cabinet Memo room 237) and hydrogen peroxide (H₂O₂ at 4°C Memo's fridge room 238), supplied by SAME.

7. Method complexity

Hazardous products manipulation: nitric acid and H₂O₂, micro-wave.

8. Procedure

Weight around 5mg (for seeds) and 20mg (roots, leaves) of dry material.

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

Transfer the powder in Quartz tubes (supplied by SAME in a crystal box room 237).

I Preparation : *all steps under the fume hood.*

Add 750µl of Nitric Acid 65%

Add 250µl of H₂O₂ 30%

Gently homogenize.

Let the tubes sit 10 minutes under the hood.

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Meanwhile:

- 1- switch on the microwave (green button on the left side)
- 2- switch on the controller (orange button)

Login : TSF

Password : tsf

Choose the program «COURT_180108 » about 1h10;

Or « LONG_180108 » about 1h30.

See with Sandrine CHAY



II Closing the system :

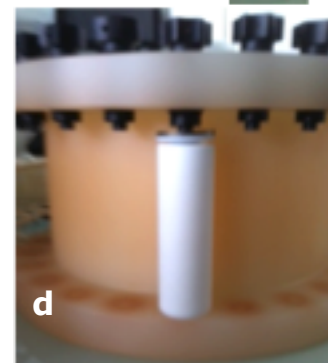
- 1- Press together the 3 parts of the cap (a) using the conical tool (b)



- 2- Place the quartz tubes in the corresponding teflon adapter and place the cap (c)



- 3- Screw the tubes on the teflon rotor (d)



- 4- Equilibrate the tubes (note the tube numbers on the rotor)

- 5- Screw tightly the tubes using the tool (e)

III Starting-up:

- 1- Place the rotor in the basket
- 2- Put the rotor-basket in the microwave
- 3- Screw the exhaust tube in the basket
- 4- Start the program « Start » 2 times

IV End of the Run : *Wait 30 minutes after the end of the run before to open the microwave and to remove the basket. Further handling must be done under the fume hood.*

- 1- Unscrew the exhaust tube

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- 2- Remove the basket and place it under the fume hood
- 3- Gently take the rotor out of the basket
- 4- Unscrew the caps
- 5- ***If the samples are transparent*** (sample completely digested, no particles remaining)
=> withdraw yours samples from the tubes

If the digestion if not complete: make a second RUN

V for the subsequent elemental analyses (MP-AES):

Dilute the samples in order to reach a final HNO_3 concentration between 5 -10% (corresponds to a 1/5 dilution in MilliQ water)

Transfer samples in the convenient tubes for the autosampler (Agilent PPP tubes supplied by SAME room 238).

The samples can be stored in the cold chamber.

NB: For the auto sampler, the void volume is approx. 700 μl .

To analyze 4 elements, make sure you have at least 800 μl of liquid + void volume.

VI cleaning and maintainance:

- 1- Put the 3 parts of the caps in a « crystal box » with distilled water.
- 2- Wash the quartz tubes with distilled water several times. Keep them on the rack during the cleaning, use a bottle brush. For the last rince use MilliQ Water.
Caution ! Handle with care, these tubes are fragile and very expensive !(about 195€ each)
- 3- Let the tubes dry bottom-up on their rack.
- 4- Tubes can be dried quickly with a hair dryer if several series of samples have to be digested.

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