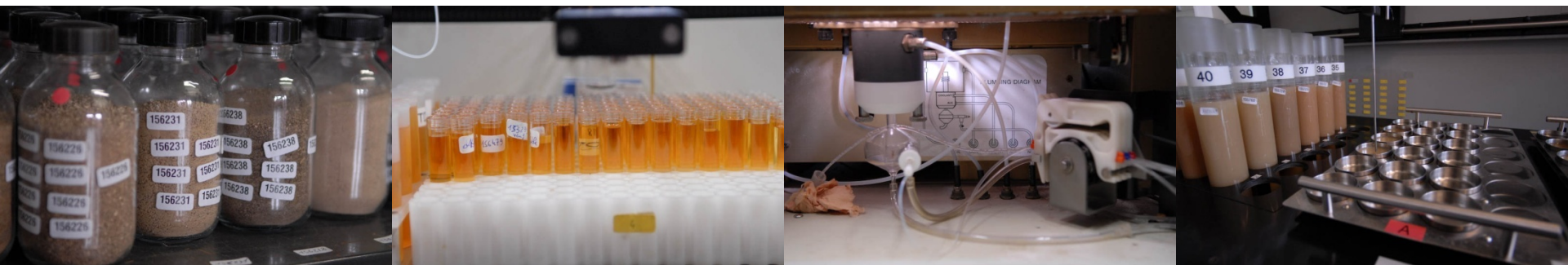


# Meeting of the Heads of the Laboratories

Warsaw, 12-13 October 2009



Soil Analysis Laboratory  
Nicolas Proix

ALIMENTATION

AGRICULTURE

ENVIRONNEMENT



# Application of microwave assisted Aqua Regia extraction to soil samples: advantages and disadvantages

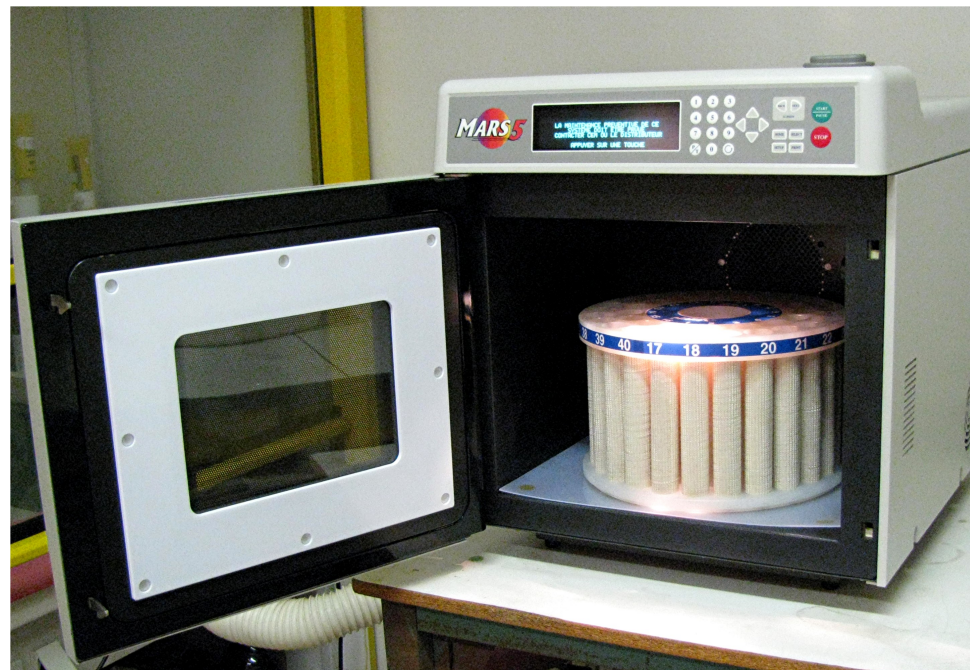


# INTRODUCTION

- Evaluate advantages and disadvantages of microwave assisted Aqua Regia extraction
- Compare with reflux aqua regia extraction method
- Underline the influence of particle size on extract element

# Extraction Method

- Microwave assisted extraction (WD ISO TC 190 SC 3 WG 1)
  - 300 mg soil sample
  - 3 ml HCl, 1 ml HNO<sub>3</sub>
  - 175°C 10 min
  - Filtration, final volume 100ml
  - ICPAES or ICPMS



# Advantages

- Microwave assisted extraction (WD ISO TC 190 SC 3 WG 1)
  - Faster, and safer than reflux AQ extraction,
  - 40 samples by batch
  - No dilution need for ICPAES measurements,
  - Fewer acid consumption,
  - Less corrosion problems

# Constraints

- The low test portion level (300mg) has two consequences:
  - It becomes mandatory to use the test sample with a particle size  $< 250\mu\text{m}$ , in order to insure the homogeneity of test portion;
  - the low test portion returns a high contamination level in case of accidental contamination.

# Constraints

– In order to evaluate the consequence of changes in extraction mode, we investigate:

- Reflux AQ particle size 2mm **AQ2000**
- Reflux AQ particle size 250µm **AQ250**
- Assisted µwave AQ, particle size 250µm **AQµwaves**.

– And the following elements:

Al, Ca, Fe, Mg, Mn, Na, P, S by ICPAES

Cu, Cr, Ni, Pb, Zn by ICPAES

K Flame emission, Cd ICPMS



# Extraction Methods

- Aqua regia extraction ISO 11466
  - 3g soil sample particle size  $< 2\text{mm}$  or particle size  $< 250\mu\text{m}$
  - 21 ml HCl, 7 ml HNO<sub>3</sub>
  - Reflux 2 hours
  - Filtration, final volume 100ml



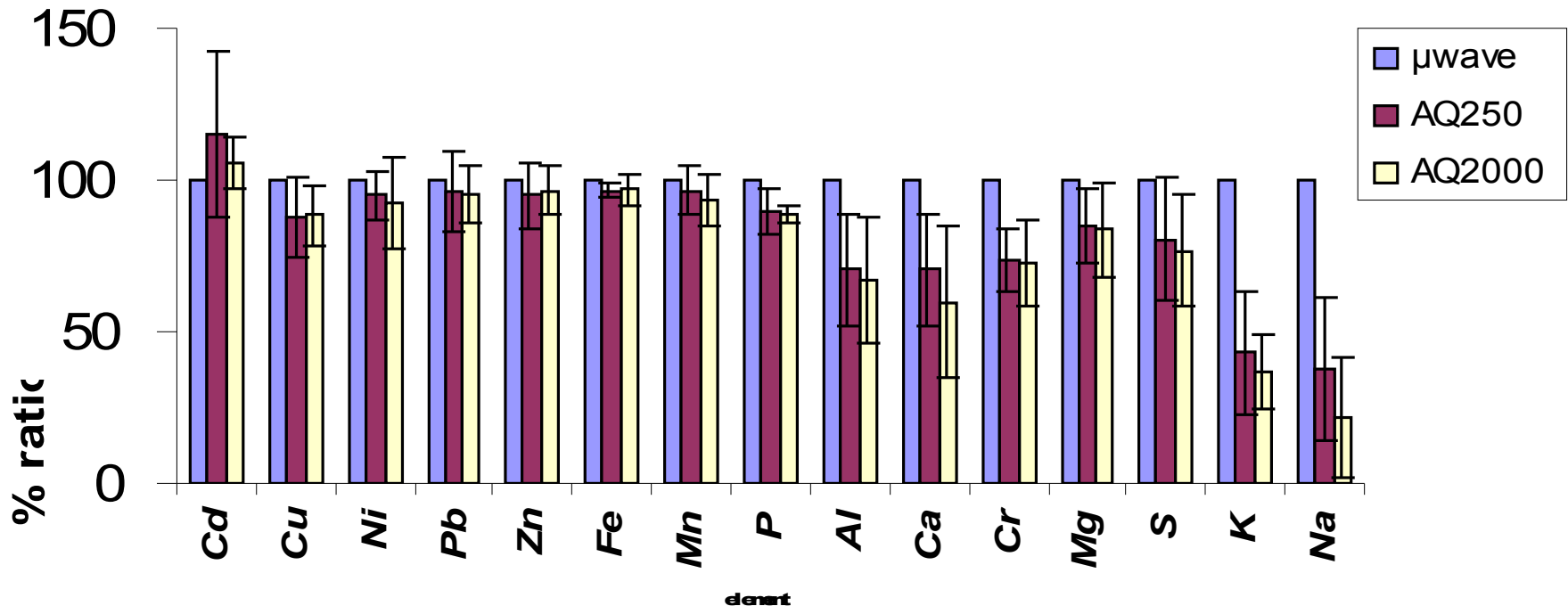


# Experimental protocol

- Selected soils:
  - Samples A, B, C from 6<sup>th</sup> FSCC Interlaboratory comparison
  - Two internal reference soils from SAL
- Protocol:
  - 3 independent extractions including 5 tests on each soil with AQ 2000, AQ 250 and AQμwaves.
- Calculation: normalization with AQμwaves value
  - $\text{value (AQ250) or (AQ2000)/value(AQ}\mu\text{wave)} \times 100$

# Results

**Ratio between reflux AQ and  $\mu$ wave AQ extraction**  
**Mean value obtained on studied soils**



# Conclusions

REF	% ratio
Cd	85-110
Cu	85-110
Ni	85-110
Pb	85-110
Zn	85-110
Fe	85-110
Mn	85-110
P	85-110
Al	70-85
Ca	70-85
Cr	70-85
Mg	70-85
S	70-85
K	< 50
Na	< 50

The assisted  $\mu$ waves AQ extraction is useful and safer than reflux AQ. The obtained results between both methods for green elements are in good agreement.

A soft shift is observed for the orange elements, and a hard shift for the red elements.

This shift is not only due to change in test sample particle size, but to more energetic extraction conditons.