Meeting of the Heads of the Laboratories
Warsaw, 12-13 October 2009

Soil Analysis Laboratory
Nicolas Proix
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 HNO₃
  - 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
The low test portion level (300mg) has two consequences:

- It becomes mandatory to use the test sample with a particle size < 250µm, in order to insure the homogeneity of test portion;
- the low test portion returns a high contamination level in case of accidental contamination.
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 < 2mm or particle size < 250µm
  - 21 ml HCl, 7 ml HNO3
  - Reflux 2 hours
  - Filtration, final volume 100ml
Experimental protocol

– Selected soils:
  • Samples A, B, C from 6\textsuperscript{th} 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
  • value (AQ250) or (AQ2000)/value(AQμwave) x 100
Results

Ratio between reflux AQ and µwave AQ extraction
Mean value obtained on studied soils

Soil Analysis Laboratory
Nicolas Proix
Conclusions

The assisted μwaves 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 conditions.