Total major and trace elements in soil obtained with an automated digestion system

Meetings of the Heads of the Laboratories – ICP Forests

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Total digestion of soils in LAS-INRA

- NF X 31-147 (French standard)

- Method:
  - 5ml HF + 1.5ml HClO₄
  - Hot block (120 °C and 160 °C, ≈7 hours)
  - Final medium: diluted HNO₃
  - 0.25 g of ashed soil / final vol: 50 ml

- Manual addition of very dangerous acids

- Time-consuming
VULCAN (Questron Technologies Corp)

Safety: only put bottles of acids in a box

Availability of technicians
Automated method of digestion

- **Steps of the program:**
  - Addition of HF and HClO₄
  - Acid evaporation
  - Vol adjusted to 50 ml with HNO₃
  - Transferring into a tube

- **Problems:**
  - Max volume transferable is 35 ml (not enough for the analysis of all the elements)
  - Blanks too high (> quantification limits)

- **New program:**
  - End = addition of diluted HNO₃ with a final vol of 30 ml
  - Steps of stirring, transferring and adjusting the vol to 50 ml are done manually
Question:

LAS is accredited for total analysis of soil
Analysis of monitoring experiments
Total digestion of soils

- **Soil samples:**
  Soils from the International Soil-Analytical Exchange (ISE, Wageningen evaluating programs for analytical laboratories)

- **Experiments:**
  - Automated digestion of 56 soils, 1 replicat / soil
  - Automated digestion of 8 soils, 2 replicats / soil, repeated 5 times
    - comparison of results: automated method versus manual method (data from 2014 to 2017)
    - calculation of z-scores
    - accuracy profils (NF V 03-110)
    - estimation of uncertainties
Analytical methods

ICP-MS
As - Cd, Mo, Pb, Tl

ICP-AES (axial)
Co, Cr, Cu, Ni, Pb, Zn - S

ICP-AES (radial)
Al, Ca, Fe, K, Mg, Mn, Na, P
Results: exemples of Fe and As

$y = 0.9985x$
$R^2 = 0.9965$

$y = 0.9917x$
$R^2 = 0.9974$

⇒ « Automated » and « manual » results seem to be not significantly different.
Results: exemples of K and Cr

⇒ Open digestion systems (manual and automated) induce variable losses of Cr

⇒ Analytical problem suspected
Results: number of z-scores ≥ 3

⇒ K: analytical results must be checked
⇒ Soil 856: 5 outliers (Ca, Cd, K, P, Zn) → problem of digestion
⇒ Cr: only 1 outlier, despite variability (large standard deviation of ISE)
Results: Accuracy profil and uncertainty

Accuracy profil of Fe

⇒ Expanded uncertainty (k=2) : 5%
Conclusion

First results:
- Concentrations obtained with automated digestion are globally in accordance with previous results (manual digestion method)
- Conform results for Fe
- Some problems:
  - K : truness
  - Cr : precision

Next steps:
- Finish exploitation of the results
- Check K results
- Present results and conclusions to the COFRAC (French comitee of accreditation)
Thank you for your attention
## LOQ Total HF digestion of soil

<table>
<thead>
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<th>Method</th>
<th>LOQ (g/100g)</th>
<th>Unity</th>
<th>LOQ (mg/l)</th>
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