Outcome & Feedback from the 6th Meeting of the Heads of the Laboratories
Solved and new tasks for the EP Meetings

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ICP-FORESTS - Combined Expert Panel meeting

Brussels/Belgium
6th Meeting of the Heads of the Laboratories
7.-8. September 2017 in Verbania-Pallanza

33 participants from 17 countries
Drying of Foliage samples - Manual part XII
(Task from EP Meeting Zagreb)

Replace:
➢ “Only the total concentration of elements in needles or leaves must be
given by reference to 105°C-dried material (Table 2).”

With:
➢ “The total concentration of elements in needles or leaves and dry mass
of 100 leaves or 1000 needles must be given by reference to 105°C-
dried material (Table 2).”
Drying of Litterfall samples - Manual part XIII
(Task from EP Meeting Zagreb)

- Dry weight [kg/m²] (total, foliage, other biomass) → dried at 70°C!
- Dry mass [g] of 100 leaves or 1000 needles → dried at 70°C?
- Results [concentration] of the chemical analysis → corrected to an oven-dry mass of 105°C!

Inaccuracy in calculating the input of elements (~5% too high)
Drying of Litterfall samples - Manual part XIII
(Task from EP Meeting Zagreb)

- Dry weight 70°C [kg/m²] (total, foliage, other biomass) $dry\_weight\_70$
- Dry weight 105°C [kg/m²] (total, foliage, other biomass) $dry\_weight\_105$
- Dry mass [g] of 100 leaves or 1000 needles at 105°C (specify in the manual & database)
Add heavy metals in the - Manual part XII and XIII (Task from lab heads meeting)

- The new parameters As, Cr, Co, Hg and Ni should be added during the Manual update 2020 in the foliage and litterfall manuals.
## Changes in the method code list

<table>
<thead>
<tr>
<th>Code list</th>
<th>FO/LF/GB</th>
<th>SO</th>
<th>DE/SS</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Element analyzer</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DA01 Macro Elemental-analyzers for C, N or S total for solids (Sample &gt; 100mg)</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>DA02 Micro Elemental-analyzers for C, N or S total for solids (Sample &lt; 100mg) with an extra milling step</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>DA03 TOC/TN Analyzers (total organic C/total N-Analyzer) for liquids</td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>DA04 CO$_2$-Determination, Calcimeter (Scheibler unit)</td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>DA05 Hg-Analyzer</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>DA06 Macro Elemental-analyzers for C for solids with variable temperature-programming for Corg- and CO$_3^-$-determination</td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>DA07 Macro Elemental-analyzers for C for solids with CO$_3^-$-unit or automated CO$_3^-$-analyzers (acid digestion of carbonates)</td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>DA08 Macro Elemental-analyzers for C for solids; Corg-determination after acid Carbonate-degassing; CO$_3^-$-determination as difference between Ctot and Corg</td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>DA99 Other Element analyzers methods</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
</tbody>
</table>
Which system do you use for aqua regia extract?

- Reflux system with quarz glas tubes and glas reflux condenser – 16 labs
- Reflux system with Teflon tubes and teflon reflux condenser – 3 labs
- Microwave system – 15 labs
- Without reflux – 1 lab

Comparison between reflux method and microwave extraction with aqua regia was planned in the 9th Soil Ring Test
9th Soil Ring Test 2018/19

- One webinterface for normal ringtest evaluation (& reflux method)
- One webinterface only for data collection from the microwave extraction method

Only two laboratories submit results (only) from the microwave extraction method! ✗

Probably more laboratories use the microwave extraction method and submit the code from the reflux method ✗
Microwave extraction method

New method codes:

- **PD06** Microwave extraction method (EN 16174)
- **PD07** Microwave extraction method (ISO 12914)
- **PD08** Microwave extraction method (not specified)

**Questionnaire (till the labheads meeting):**
- Digestion temperature/pressure/time
- Acid mixture
- Sample weight
- Instrumentation

**Method harmonization**
Evaluation limits for Al, Fe and Mn were fixed in Pallanza 2017:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Conc. Range low</th>
<th>Tolerable Limit low</th>
<th>Conc. Range high</th>
<th>Tolerable Limit high</th>
<th>max. accept. LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium</td>
<td>mg/l</td>
<td>( \leq 0.1 )</td>
<td>( \pm 30% )</td>
<td>( &gt; 0.1 )</td>
<td>( \pm 15% )</td>
<td>0.05</td>
</tr>
<tr>
<td>Iron</td>
<td>mg/l</td>
<td></td>
<td>( \pm 30% )</td>
<td></td>
<td>( \pm 30% )</td>
<td>0.02</td>
</tr>
<tr>
<td>Manganese</td>
<td>mg/l</td>
<td>( \leq 0.025 )</td>
<td>( \pm 15% )</td>
<td>( &gt; 0.025 )</td>
<td>( \pm 10% )</td>
<td>0.01</td>
</tr>
</tbody>
</table>
Deposition / Soil Solution – Heavy Metals in Ring Test & Monitoring

- Deposition/Soil solution: Cd, Cr, Co, Cu, Pb, Ni, Zn were included in the ring test program.
### Heavy Metals - evaluation limits

**Atmospheric Deposition and Soil Solution Working Ring test**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Conc. Range low</th>
<th>Tolerable Limit low(^*))</th>
<th>Conc. Range high</th>
<th>Tolerable Limit high(^*))</th>
<th>max, LOQ(^**)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cadmium</td>
<td>µg/l</td>
<td>≤ 1</td>
<td>± 40 %</td>
<td>&gt; 1</td>
<td>± 30%</td>
<td>0.1</td>
</tr>
<tr>
<td>Cobalt</td>
<td>µg/l</td>
<td>≤ 1</td>
<td>± 40 %</td>
<td>&gt; 1</td>
<td>± 30%</td>
<td>0.1</td>
</tr>
<tr>
<td>Chromium</td>
<td>µg/l</td>
<td>≤ 1</td>
<td>± 40 %</td>
<td>&gt; 1</td>
<td>± 20%</td>
<td>0.5</td>
</tr>
<tr>
<td>Copper</td>
<td>µg/l</td>
<td>≤ 2</td>
<td>± 40 %</td>
<td>&gt; 2</td>
<td>± 20 %</td>
<td>1</td>
</tr>
<tr>
<td>Nickel</td>
<td>µg/l</td>
<td>≤ 1</td>
<td>± 40 %</td>
<td>&gt; 1</td>
<td>± 20 %</td>
<td>0.5</td>
</tr>
<tr>
<td>Lead</td>
<td>µg/l</td>
<td>≤ 1</td>
<td>± 40 %</td>
<td>&gt; 1</td>
<td>± 25 %</td>
<td>0.5</td>
</tr>
<tr>
<td>Zinc</td>
<td>µg/l</td>
<td>≤ 30</td>
<td>± 35 %</td>
<td>&gt; 30</td>
<td>± 25 %</td>
<td>10</td>
</tr>
</tbody>
</table>

*\(^*)\) Concentration limit
\(^**\) Maximum Limit of Quality

**Mandate from the EP Depo to the next labheads meeting**

\[X\]
Deposition – skip parameters
(Task from lab heads meeting)

- All parameters (laboratories) of the “chemical surveys” had to be tested with ring tests so:

- The meeting of the heads of the labs proposed to the expert panel deposition to **skip Hg, B and Mo** from the parameter list, because of methodical problems and this parameters are not and can not be analyzed in the regular ICP-FORESTS DP survey. This could be done during the next manual update 2020.
<table>
<thead>
<tr>
<th>Ringtest</th>
<th>Monitoring survey interval</th>
</tr>
</thead>
<tbody>
<tr>
<td>Foliage and Litterfall annually</td>
<td>Foliage every 2 years, Litterfall annually</td>
</tr>
<tr>
<td>Soil every 3 years</td>
<td>Soil &gt; 10 years</td>
</tr>
<tr>
<td>Deposition &amp; Soil Solution every 2 years</td>
<td>Deposition &amp; Soil Solution continuous</td>
</tr>
</tbody>
</table>

*Frequency of ringtests have a positive influence on the data quality!*
Deposition & Soil Solution

Ring Tests

- Should be made more often – once per year!
- First proposal was presented yesterday
- Final draft will be evaluated till the next meeting of the heads of the labs

Must be adopted in the EP Meeting and in the labhead meeting ❌
QA/QC Manual Update

- When a (ICP-Forests) lab did not qualify and did not make efforts to requalify, the ring test organizers ICP Forests PCC will send a letter to the National Focal Centre and inform them about the consequence that their data possibly cannot be used for evaluations on an European level.

- The results of the ring tests are integrated in the database of the PCC. This means that the bad ring test results will be marked as disqualified and this information can be used as a selection criterion for the monitoring data used in evaluations.
7th Meeting of the Heads of the Laboratories is scheduled in Brasov/Romania

5.9. – 6.9.2019

Details will follow soon…!