ICP-Forests Combined meeting of the Expert Panels Foliar and Litterfall, Deposition, Soil/Soil Solution, Crown Condition and Damage Causes, Ambient Air Quality together with the Working Group QA/QC in Laboratories – 9 March 2021

Minutes

(All presentations can be downloaded from the ICP Forests website: [http://icp-forests.net/group/qualityinlaboratories/page/document-archive](http://icp-forests.net/group/qualityinlaboratories/page/document-archive))

A. Meeting of the Working Group QA/QC in Laboratories 9. March 2021

Mr. Alfred Fürst opened the meeting thanked the PCC for organizing this virtual meeting and welcomed the participants. The agenda was adopted.

Mr. Alfred Fürst presented the planned Workplan 2021/22 under influence from Covid-19. The **24th Needle/Leaf Interlaboratory Comparison Test 2021/2022** will be performed as planned. At the moment the registration is open till 25th June 2021 (at the moment 22 participants). See: [https://bfw.ac.at/ws/ring_nadel.login](https://bfw.ac.at/ws/ring_nadel.login) responsible up from now is Michael Tatzber (michael.tatzber@bfw.gv.at).

The **11th Atmospheric deposition and soil solution Working Ringtest** is postponed to 2022, and is valid for 2021 & 2022. The planned schedule is:

- Invitation - end of August 2021
- Registration - end of September 2021
- Sending samples - end of October/beginning of November 2021
- Submission of the results - end of February/beginning of March 2022
- Requalification - beginning of April 2022

The planned schedule for the **10th Soil Interlaboratory Test 2021** is:

- Registration till 15th April 2021
- Sample submission till April 2021
- Data submission till 1st October 2021
- Evaluation in October 2021
- Requalification ends April 2022
All ringtest results can be discussed at the next meeting of the heads of the laboratories. This meeting is postponed and will be held in May 2022 – hopefully as real meeting – in Switzerland at WSL/Birmensdorf. A detailed agenda will be provided end of 2021. Mr. Fürst thanked all Swiss colleagues which are/will be involved in organizing this meeting.

Alfred Fürst informs the participants that Anna Kowalska will take over the chair of the WG-QA/QC next year (Heads of the laboratories Meeting/TF Meeting 2022), so we are searching for a new co-chair for the WG-QA/QC in laboratories.

Ms. Anna Kowalska presented How to proceed with laboratories which failed in the ringtest and show no improvement? Some laboratories that participate in the ICP-Forests ringtests fail for one or more parameters and show no improvement with time. They get requalification and in the next ringtest(s) fail again for the same parameter. There is a need to implement a procedure that would motivate those labs to make progress.

Manual Part XVI version 2020 provides that “When a lab did not qualify and did not make efforts to improve the data quality, ICP Forests PCC will send a letter to the NFC and inform them about the consequence that their data possibly cannot be used for evaluations on an European level”. In details the procedure would consist of the following steps:

1. If the lab passes the ringtest – the results are accepted without further steps for survey year covered by the period of validity of the ringtest.

1a. If the lab fails and needs a requalification for a parameter in ringtest N - PCC will send a letter to NFC informing that monitoring data from the survey year N will be marked as possibly wrong for that specific parameter, if the lab failes in the next test (N+1) too, or if does not take part in the next ringtest.

2. If the lab passes the next ringtest (N+1) – monitoring data for years covered by ringtests N and N+1 are accepted without further steps.

2a. If the lab fails in the ringtest N+1 - monitoring data for year covered by ringtest N (for that specific parameter) are marked as wrong in the database. If the lab passes the requalification in ringtest N+1 then PCC will send a letter to NFC informing that monitoring data from the survey year N+1 (for that specific parameter) are marked as wrong, if the lab fails in the next ringtest again.

Monitoring results should be better linked to the quality information from the ringtests (see presentation on Monday from Manual Nicolas - on the activities of the Quality Assurance Committee and to the following presentation of Till Kirchner). A fast feedback about unsuccessful ringtest participation must be given as fast as possible to PCC, the NFCs and concerned laboratories. The decision on pass/fail requalification is made by the ring test provider based on the plausibility check of the submitted documents of the laboratories. The effectiveness of the measures against bad data quality can only be proved, if the laboratory takes part in the following ringtest and passes immediately for this parameter. Therefore, the re-qualification process is only completed after positive participation (for this lab & parameter) in the following test.

The validity period for foliar and deposition ringtests covers usually 1 year while for soil ringtest - 3 years. The feedback from the next soil ringtest comes 3 years later. The possibility of organizing soil ringtest more often should be discussed within the EP Soil.
In the following discussion (Mr. Bruno de Vos, Ms. Nathalie Cools, Ms. Tamara Jakovljević) there were some ideas how to handle this topic for the soil tests: shorten the ringtest repetition period (two years) or submit an extra reference sample set (old test samples) to check against the same error in future. It was agreed to discuss this in the following EP Soil.

**Mr. Bruno de Vos** proposes to establish guidelines that new laboratories must follow before participating in a ring test for the first time. Mr. Alfred Fürst refers to the QA/QC in labs manual and will try to compile a simple guideline with corresponding literature until the next lab heads' meeting.

**Mr. Hans-Peter Dietrich** asked, if the lab helping program is still active? Ms. Anna Kowalska answered yes, but without funding. One part of the lab visits must be paid.

**Mr. Till Kirchner** presented: Link between monitoring data, data checks and Ringtest results? Quality information is currently located in various places in the database. The aim is to make this information easily accessible to data users so that data quality can also be used as a selection criterion for evaluations.

For this purpose, the monitoring data in the DEM, FOM, LFM, SOM and SSM files must be linked with data from the LQA files (e.g. method codes, LOQ) and with the ring test results coming from the test providers (e.g. labcodes, percentage ringtest result). This is not so easy, because of the long key an extra generated code number (QIF) is introduced in the monitoring data tables. This number can be used to link ring test data and LQA data as well as other quality information (e.g. % data completeness,...). Other data checks e.g. ion-balance, conductivity checks must be stored with in the extended DEM, FOM, LFM, SOM and SSM files.

However, there are of course some problems in the details, but Mr. Till Kirchner hope that the finished concept will be ready to present at the next lab heads’ meeting.

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**Mr. Michael Tatzber** presented the results of the 22nd and 23rd Needle Leaf Interlaboratory Comparison Tests. The number of participating laboratories decreased slightly compared to previous tests.

Following samples were analysed in these two tests: Spruce needles, ash leaves, Quercus robur and spruce needles in the 22nd test and beech leaves, two samples consisting of spruce needles and ash leaves in the 23rd test. Sample 1 of the 19th and Sample 2 of the 23rd Interlaboratory Comparison Test were identical (spruce needles) and their determined values in a satisfying agreement. The most prominent pre-treatment methods were microwave digestion and pressure digestion; the most prominent analytical methods were ICP-AES and ICP-MS.

The five rather new parameters arsenic (analysed by 12 labs), cobalt (14 labs), chromium (23 labs), mercury (15 labs) and nickel (25 labs) had enough participants for a statistical evaluation. For determinations such parameters ICP-MS appears as the best choice – for mercury the element analyser as well.
The results for nitrogen and sulfur could be improved; the result for calcium was not improved. It is assumed that the most prominent error sources for Ca-analyses are application of ICP-MS in a too high concentration and no right buffering against P and Si in the AAS-flame technique, also matrix adapted standards appear important for this method (with focus on acid mixture & concentration). Relatively high numbers of non-tolerable results for lead, arsenic and cobalt are in most cases originating from other used analyse-methods than ICP-MS, so this appears clearly as the best choice for analyses of such parameters.

The registration for the 24th is still open until 1 July 2021, the actual number of participants is 25 (March 2021). There is a need for samples, especially (but not only) heavy metal polluted ones and/or samples from deciduous trees! Please contact Mr. Alfred Fürst if you want to prepare a ringtest sample and keep in mind that the opportunity for its further use as a control sample is a benefit!

Ms. Tamara Jakovljević presented the 10th Soil Ring test preparation and review of the 9th Soil Ring test. Laboratory for physically and chemical analysis, Croatian Forest Research Institute will organize the 10th soil interlaboratory comparison in 2021. Registration is open till 15th April 2021. The samples A-E are from Slovenia (A), Turkey (B), Austria (C), Croatia (D) and Latvia (E). The organization includes: preparations for the registration of participants, pre-treatment, homogenization and homogeneity comparison. One sample is with CaCO₃ content between 10 – 20% and one sample is organic. The submission of the results is from May to 1st October 2021. The first results will be available in October 2021.

According to the new version of ICP Forests manual on Soil 2020, two methods for the preparation for the analyses of the semi-total elements in aqua regia extract are allowed, the reflux method and the microwave digestion method. Though, for the ringtest the laboratory should used the same method as it is done for the monitoring samples which are reported to the PCC database. If labs have the possibility to analyse the aqua regia extract with a second method (not normally used for the monitoring samples), they can submitted these results as an Excel file for a second external evaluation. This extra evaluation will be done outside of the normal ringtest evaluation.

In 9th Soil ring tests 29 labs participated. 23 labs of 29 failed at least in one parameter (mandatory or optional). 16 labs out of 23 failed one or more mandatory parameter. The most successful parameters by all labs were pH- CaCl₂, K exch and N tot. Most failed parameter was Mg extra. Re-qualification started in November 2018 and lasted till 14th June 2019 with two weeks of extension. 12 labs out of 23 submitted re-qualification results. The results were submitted to ICP Forests data base.

In case of a negative re-qualification and/or in case of negative results for the same parameter in the 10th Interlaboratory Comparison Test, the affected data of the European Level II monitoring of your laboratory will be marked due to possible poor data quality and can thus be excluded from further evaluations.
Results of the 10th Deposition & Soil solution Ring test 2020 (Anna Kowalska)

37 laboratories registered to the 10th Deposition and Soil Solution Ring test, 35 labs from 22 countries submitted results. Five of the samples were natural waters (samples 1-5): bulk open field, throughfall from Poland, stemflow (beech) from Belgium, and two soil solutions from coniferous stands in Poland. Sixth Sample was synthetic for alkalinity and phosphate measurements. Additionally 20 labs received 4 samples of natural water (samples 7-10) for heavy metals: Cd, Cu, Co, Cr, Ni, Pb, Zn. The recommended method for analysis of heavy metals is ICP-MS.

Alkalinity in soil solutions (sample 4 and 5) was not evaluated due to low pH. Phosphates in samples 1, 3, 4, ammonium in samples 4 and 5, nitrates in sample 5, Fe in sample 1, and Cr in sample 7 were excluded from the evaluation due to too low concentration.

The overall score of the labs slightly improved compared with previous rounds of ringtests: the groups of labs that deliver above 90% is more numerous than in 9th ringtest (20 and 15, respectively) and less labs delivered below 80% of correct results than before.

Requalification was necessary for 22 labs, 10 of them failed for 3 to 7 parameters; 17 of the labs made the effort to requalify bad results. Part of the problems with analysis of the ringtest samples for ringtest or for requalification can be ascribed to pandemic restrictions, mainly prolonged lockdown. However, most of the reasons for bad results do not change over the consecutive rounds of ringtests.

The most problematic parameters were SO4-S, pH and PO4-P with 5 to 7 labs failing to qualify.

Percentage of non tolerable results is comparable with two previous rounds of ring tests (2017 and 2019). For all the parameters no more than 20% results falls outside the tolerable limits.

Next 11th Deposition and Soil Solution ring test is planned to start in summer/autumn 2021 and finish in spring 2022.

Mr. Alfred Fürst: Miscellaneous:

Mr. Manuel Nicolas and Ms. Sue Benham want a better information on the current status of the individual ring tests and about the status of the re-qualification. Mr. Alfred Fürst refers to the website of the ICP-Forests (http://icp-forests.net/group/qualityinlaboratories) and to the fact that in the future the NFCs have to be informed automatically via PCC if the laboratories need to be re-qualified. In addition, the deadlines for the current ring tests will be sent out to the NFCs and to the Experts (Foliage/Litterfall/Soil/Soil solution/Deposition) once per year. In addition these links will be added to the ringtest pages on the ICP-Forests webpage:

**Needle/Leaf Interlaboratory Test**


Status of the Qualification: https://bfw.ac.at/ws/ring_nadel.qa_overview

**Deposition Ringtest:**

Status of the Qualification: [https://bfw.ac.at/depo/ring_depo.qa_overview](https://bfw.ac.at/depo/ring_depo.qa_overview)

**Soil Ringtest:**


Status of the Qualification: [https://bfw.ac.at/fscc/ring_boden.qa_overview](https://bfw.ac.at/fscc/ring_boden.qa_overview)

The person responsible for the laboratory has access to the input web interface of the ring tests - if this access is needed by the NFC, he/she must be contacted directly or if he/she agrees the password can be shared with the NFC.

Mr. Alfred Fürst closed the meeting.