

History and progress of the ICP Forests ringtest programme and the Working Group QA/QC in Laboratories

Implementation of laboratory comparison tests (ringtests) and a web interface for data submission

An important - and mostly too little considered - factor in long-term environmental monitoring is consistently good data quality in the laboratory. After all, only actual changes or small trends in nature should be detected - not fluctuations in the quality of laboratory data. This was taken into account at a very early stage in the ICP Forests Level I and Level II surveys. In the early 1990s before the Europe-wide soil and foliage surveys on Level I started, the Expert Panels Soil and Foliar decided to carry out laboratory comparison tests (ringtests) prior to and in parallel with their surveys. We would like to show the progress towards the implementation of the modern ring test web interface compared to earlier versions of the Needle/Leaf Interlaboratory Tests. In 1993/94, the first test was carried out using two standard reference materials provided by the BCR (European Commission). The main purpose of this first test was to eliminate unsuitable methods. At the second Expert Panel Meeting in Ås/Norway in March 1994, it was decided to continue the ring test programme in parallel with the Level I survey in 1995/1996. The evaluation of the Level I foliage data was to be carried out by the newly established Forest Foliar Co-ordinating Centre (FFCC) at the Austrian Research Centre for Forests BFW in Vienna. The ring test provider for the next four tests was the North Rhine-Westphalia State Environment Agency (LUA). The ringtests were to be conducted every two years and run in parallel with the Level II foliage surveys. During this time, the Level I and II surveys and evaluation as well as the ring tests were funded by the EU (DG Agri). Test evaluation typically took several months, as the data were sent by post or fax to the ringtest provider, entered into the evaluation program, and a printout returned to the laboratories for re-checking. Only after the correctness of all data was confirmed, the evaluation was carried out, and the report was generated and sent to the laboratories.

After the 5th test in 2001/2002, LUA could no longer continue the ring tests and they were taken over by the FFCC. The following difficulties had to be solved first:

- The evaluation time of the ring tests had to be shortened drastically so that the laboratories could react quickly to unsatisfactory results.
- A change in funding was necessary, because the EU (DG Agri) no longer funds the ICP Forests programme.

Out of these needs, the FFCC developed a web-based interface for easy and simple collection of ring test data, which is still used today. It allowed direct input of laboratory data and re-checks via the Internet and had an export function for further evaluation. The costs of the ring test were reduced and can also be covered by laboratories with a participation fee, and ringtests should be opened to other laboratories to share the cost. Ring tests are no longer tied to project funding.

The implementation of soil- (from 2007) and water ring tests (from 2009) was also switched to the WEB interface data collection and adapted by the FFCC to their specific needs.

Establishment of the Working Group QA/QC in Laboratories

In 2007, the Working Group QA/QC in Laboratories was established. The members came from the respective Expert Panels, the FFCC, and the Forest Soil Co-ordinating Centre (FSCC) of ICP Forests as well as from the laboratory working group located in the Expert Panel Deposition and contributed their experience to the new working group.

The further harmonization of the ring tests and their implementation was an essential task to be continued, building upon the developments made in the years before. When the EU (DG Env) funded the FutMon (Future Monitoring) Life+ project, which started in 2009, it was possible to obtain time and money for the improvement and implementation of the ring test programme. The next major steps were:

- Harmonization of laboratory codes between laboratories for soil, foliage, and deposition/soil solution.
- Harmonization of the method codes to be able to use them in the new LQA files as well.
- Extension of the WEB interface to include a module for standard ringtest evaluation (median-based) and for recording the results of laboratory re-qualifications.
- Creation of online qualification reports for participating laboratories.
- Ring test providers needed to be found to do the practical work of conducting the ring test (tendering the test, collecting and sending samples, invoicing participants, evaluation, etc.).
- Involve PCC and NFCs in their responsibilities to ensure sufficient quality of the monitoring data in the laboratory. This was newly included in the most recent update of the ICP Forests Manual, Part XVI “Quality Assurance and Control in Laboratories” and will be more strongly incorporated in the “Manual Part III: Quality Assurance within the ICP Forests Monitoring Programme”.
- Create a uniform data extract of the ring test results for the PCC database, and finally link ringtest results to monitoring data as quality indicator.

The implementation of the ring tests is finally a routine task; 45 tests have been conducted to date (24 foliage, 10 soil, and 11 water ringtests). Nearly 100 laboratories participate in the ICP Forests ringtest programme (see Fig.9-1).

The highest number of participants took part in the tests during the EU Life+ FutMon project (2009-2011).

All ring test reports, the current status of the qualification of the laboratories in the last test, the evaluation limits and other documents (templates for re-qualification, control charts, validation of the measured values, and links to reference material) are available on the ICP Forests homepage, depending on the different matrix (see: <http://icp-forests.net/page/working-group-on-quality>).

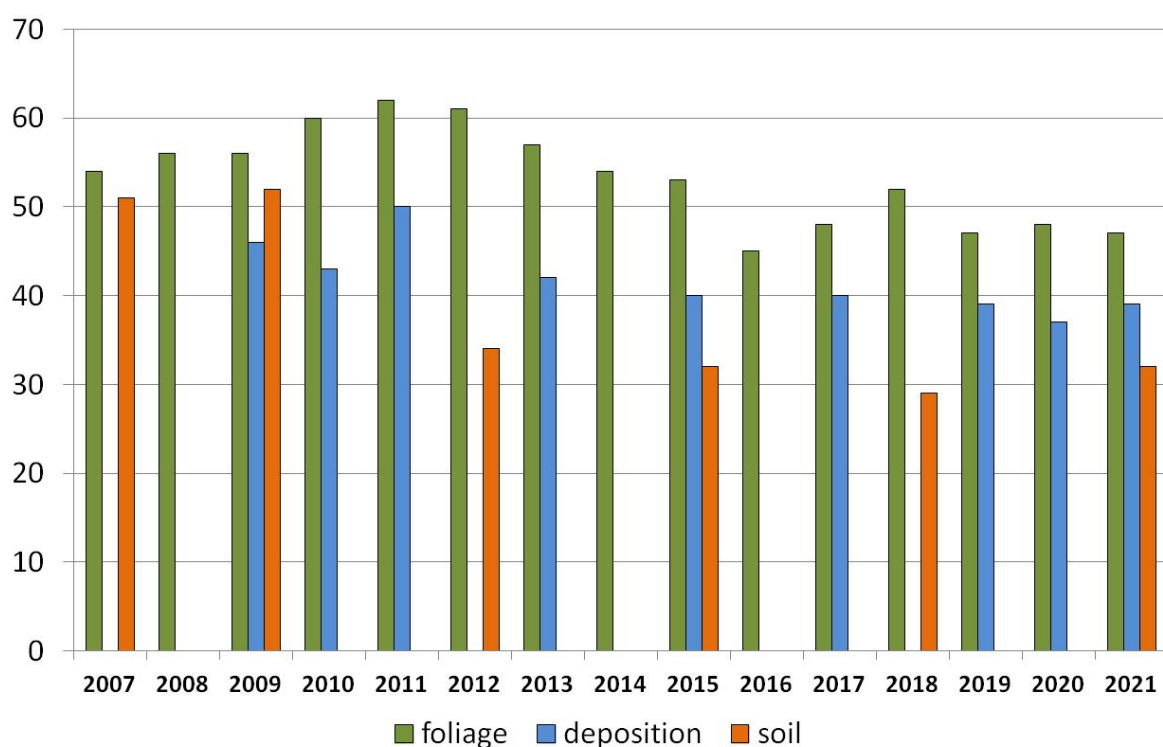


Figure 9-1: Number of participating laboratories in the ringtests from 2007 to 2021

For soil and foliage, ring test samples have also become suitable reference materials for method validation in the laboratory. Ringtests and reference materials are important tools to validate and regularly re-check the performance of analytical methods. This is particularly necessary in case of changes in staff training and experience, and of changes in equipment.

Another important task at the start of the FutMon programme was the creation of the new joint “Manual Part XVI: Quality assurance and control in laboratories”. This task was completed in May 2010; the manual has been updated twice so far in 2016 and in 2020. The most important topics in the manual deal with the use and type of reference materials, the use of control charts for lab-internal quality control, the determination and use of detection- and quantification limits, the verification and checks of analytical data, the description and procedure of ring tests, quality indicators, and reports.

Knowledge sharing was and is an important task of the WG QA/QC in laboratories. During the Forest Focus and FutMon programmes funded by the EU (DG Env and Life+ Regulation) it was possible to raise funds for on-site audits in the laboratories. This made it possible to visit the laboratories and, if problems were found, to fix them immediately or to undertake direct advisory tasks. This option was very efficient but unfortunately also expensive, so it was not possible to continue it after the funding by the EU ended. Perhaps new funded projects will arise here in the future that will allow this laboratory assistance programme to continue. In any case, direct assistance between individual laboratories is possible, as has already been done in the past, if funding is provided by the requesting laboratory.

Another way of sharing knowledge is to hold a regular meeting of the heads of the laboratories. Here, the results of the individual ring tests can be discussed and the participants have the opportunity to address problems and report on new methods. The first meeting of the Heads of the Laboratories with 37 participants took place in Hamburg

(Germany) on June 9-10, 2008 and the eighth will be held in Birmensdorf/Switzerland in May 2022. Participation in these meetings should also be more strongly encouraged by the NFCs, not only in financial terms. Personal participation in these meetings and direct contact with other colleagues provides the opportunity to discuss problems in the laboratory and hopefully find solutions, even if sometimes English is not perfectly spoken. All contributions from the meetings, as well as the minutes, are available on the ICP Forests website for further use.

To maintain the connection of the Expert Panels Deposition, Soil and Soil Solution, and Foliage and Litterfall with the Heads of the Laboratories, the WG QA/QC in Laboratories always meets together with the Expert Panels at the biennial joint Expert Panel meetings. Tasks from the Expert Panels, e.g. new parameters, LOQ, allowed or new methods, etc., can be easily passed on to the WG QA/QC and are then discussed in the Meeting of the Heads of the Laboratories. Results of the Meetings of the Heads of the Laboratories are communicated back to the individual EPs.

Collecting quality information (analytical methods, LOQ, ringtest results) and linking it to monitoring data is a new task for the PCC's database managers. The first attempts to link the ring test number and the laboratory code to the monitoring data date back to 2005 to the EP Foliage. At that time, this information was simply added to the data files. But during the harmonization of data submission, the WG QA/QC in Laboratories proposed to collect these data in a separate file (LQA-file) and to upload it to the PCC database together with the monitoring data. The ultimate goal is that each measured value for each variable can be linked to the corresponding laboratory quality indicators and ringtest result. For each individual dataset this provides information on the quality and the uncertainties of the data.

A brand new task for the database managers is the introduction of more complex data checks when uploading these LQA files to minimize data entry errors. Here, close cooperation between the Expert Panels, the WG QA/QC in Laboratories, and the ringtest providers with the database managers is important. In addition to simple range checks of the inputs, complex checks of the digestion and determination methods and their combination per parameter were developed in 2021 initially for foliage and litterfall (see example in Fig. 9-2).

Element		Element Analyser				Flame-AAS, AFS or CV					Flame less AAS		ICP-AES		ICP-MS	Other method						
		DA01	DA02	DA05	DA99	DB01	DB02	DB03	DB06	DB07	DB04	DB05	DB08	DB09	DB10	DB99	DD...	DE...	DF...	DZ02	DZ99	
As	Arsenic																					
B	Boron																					
Cd	Cadmium																					
Ca	Calcium																					
C	Carbon																					
Cr	Chromium																					
Co	Cobalt																					
Cu	Copper																					
Fe	Iron																					
Pb	Lead																					
Mg	Magnesium																					
Mn	Manganese																					
Hg	Mercury																					
Ni	Nickel																					
N	Nitrogen																					
P	Phosphorus																					
K	Potassium																					
S	Sulphur																					
Zn	Zinc																					

Legend

Valid

Warning

Error

Status: Apr 2021

Figure 9-2: Validity database check of determination methods for foliage and litterfall (see: <https://icp-forests.org/documentation/ExplanatoryItems/211.html>)

The methods coloured green in Figure 9-2 are recommended in the “Manual Part XII: Sampling and analysis of needles and leaves” and are also analytically chemically possible for this parameter and the concentration of the parameter lie within the expected concentration range. The methods coloured yellow are analytically chemically possible for this parameter and are within the expected concentration range. However, they are not explicitly listed in the manual. The methods coloured red are not suitable for this parameter. The current ring tests are a good source for finding out suitable methods; changes and the use of new methods can also be derived from the ring test results. These complex check routines were initially used for foliage and litterfall and will need to be developed for soil, soil solution, and deposition in the future.

As has been shown, quality assurance and control in laboratories is a never-ending continuous task and has to do with constant improvement and, of course, with continuous work, as the saying goes – the better is the enemy of the good.

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