

**Meeting of the Working Group QA/QC in Labs**

**12-13 September 2007, Eger, Hungary**

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**0. Introduction**

Judit Sitkey and András Szepesi welcomed the members of the WG in Eger, Hungary, and gave some technical introductions.

Nils König as the chairman thanked them both very much for the excellent preparation of the meeting and the wonderful hospitality.

**1. Plausible range checks for plant and soil analyses**

Alfred Fürst presented plausible range checks for different tree species based on level I results. It was decided to use these data as a first step. The ranges should be checked with Level II data (from JRC), and completed for other tree species with data from individual countries. There is also a need for ranges for litterfall (data from the expert panel).

Bruno de Voss presented plausible range checks for soil and humus samples and proposed some other checks: C/N ratio check, C/P ratio check, pH check, pH/CO<sub>3</sub> check, C balance check, extracted elements/total elements check, reactive elements/total elements check etc. It was decided to complete the plausible range check table with data from the BioSoil project (especially for exchangeable cations in humus) and to integrate the new check proposals into the new quality check paper (see Topic 9).

**2. Plausible range and quality checks of the data before importing into the EU data base**

Nils König presented a presentation prepared by Tracy Houston (JRC) about the data checking program of the JRC for all Level I and Level II data. However, the presentation gave no precise information about the basis of the different checks. It was decided to ask for more detailed information, especially for the plausible range checks: are they based on data from the different countries or even from different plots? Will the JRC continue the database after 2006?

**3. Improvements to the data reports: integration of ?limits of quantitation/detection limits?, means, coefficients of variation (CV %), ringtest results?**

After a short discussion it was decided to ask the JRC to integrate the following information into the datafiles for deposition, soil solution, soil and foliar analyses:

- quantification limit (QL) (for each parameter)
- detection method (coded) (for each parameter)
- coefficient of variation (%) (for each parameter, for two concentration ranges)
- coefficient of variation (%) from the yearly control chart (for each parameter, for each year)
- participation in the yearly ringtests (yes/no), ringtest number, number of the lab, ranking information (see Topic 7)

This information can be used to report the quality of the stored data, where it makes sense. It was discussed if it is possible to produce reference material (soil, foliar) for all labs in the EU (for the control charts)

**4. 9<sup>th</sup> foliar ringtest**

Alfred Fürst presented an overview of the results of the 9<sup>th</sup> foliar ringtest. In comparison with the results of the previous 8 ringtests there has been a continuous improvement. Therefore the tolerable limits (expressed as deviation of the mean in %) have been reduced in 2 steps over the last years. The reason for this improvement in quality is the training effect of the

nine repetitions of the ringtest and the use of old ring test material for method validation from the labs.

It was decided to include tolerable limits for ringtests into the manuals (see Topic 5)

#### **5. Tolerable limits for ringtests (soil, water, foliar)**

Alfred Fürst, Bruno de Vos and Rosario Mosello presented proposals for tolerable limits for the different ringtests (foliar, soil, water). After a short discussion, it was decided not to use coefficients of variation (like in the proposal from Bruno De Vos) but deviations in % from the mean (like in the proposals of Alfred Fürst and Rosario Mosello) for the definition of the tolerable limits. Two tolerable limits shall be set: one for the low concentration range and one for the normal concentration range.

Alfred Fürst, Bruno de Vos and Rosario Mosello agreed to develop new tables with tolerable limits for 2 concentration ranges for foliar, soil and water ringtests.

Due the fact that foliage concentrations are quite similar in different samples only one set of tolerable limits is used. For litterfall analyses it could be necessary to develop a second - different one - in the Expert Panel Foliage and Litterfall.

#### **6. Presentation of a cost plan for ringtests 2008-2013**

Alfred Fürst, Bruno De Vos and Rosario Mosello presented cost estimates for single foliar, soil and water ringtests. Nils König presented a cost plan for a ringtest program 2008 – 2013. This program includes meetings for the heads of the labs to discuss the ringtest results and the analytical problems. This plan was accepted. It was decided to include the costs for the assistance program for the laboratories with unacceptable ringtest results (10000 Euro/year => 5-6 laboratories) in the whole program. The cost estimate will be presented by Nils König during the ICP workshop for the FutMon project in Hamburg, Oct. 2007.

John Derome agreed to ask colleagues from DG Environment (Schulte, Rautio) about what is the best way to obtain money for this program: as a single project in Life+, integrated in the FutMon project or as a COST project.

#### **7. Consequences for a lab/NFC after bad ringtest results**

After a longer discussion it was decided to provide labs with bad results with positive encouragement, and not to put any pressure on them.

1. step: after a ringtest each participant (and each NFC) will receive a qualification report from the WG (for each parameter within the tolerable limit)
2. step: laboratories with parameters without a qualification shall be given the opportunity for requalification (reanalysis of the ringtest samples, report to the WG with the new results and the reasons for the bad results during the first analyses); after the report, the laboratory (and the NFC) will receive a requalification report.
3. step: the results of the ringtest are integrated in the data reports to JRC; this means that bad ringtest results will be known and it can be used as a criterion for rejecting the data before being used in evaluations.

#### **8. Information about the new Quality Committee of ICP Forests and related tasks for the WG and the EP**

Nils König presented a presentation of Marco Ferretti (chairman of the new quality committee) about the proposed work of the new quality committee. The WG agreed with the proposed new structure for the manuals. Specific comments to the presentation of Marco Ferretti should be sent by each member of the WG directly to Marco Ferretti.

Nils König shall send the proposed ringtest program as a part of the QA/QC program of ICP Forests to Marco Ferretti. The quality indicators developed by the deposition expert panel and the WG QA/QC should be integrated into the report of the quality committee.

#### **9. Preparation of a paper for Labs/NFC`s with all necessary quality checks**

Nils König presented a proposal for the structure of the new quality check paper for Labs and NFC`s. It was decided to prepare a draft of the paper by the end of December 2007

(extended version for the labs, later a short version for the NFC's) and to divide the work between the members of the WG (see annex). Changes to the structure are:

- chapter 3 will have 3 subchapters for water, soil and foliar
- a new chapter "contamination problems" will be included

### **10. Assistance program for labs with unacceptable results in ringtests**

Anna Kowalska and Nils König gave short reports about their assistance activities for labs in Russia (2) and Slovakia. It was the overall opinion of the members of the WG that this assistance program is considerably improving the quality in those labs which have received help. Therefore it was decided to continue the program and to broaden it to labs engaged in soil and foliar analyses.

A list of laboratories with satisfactory results will be produced from the results of the individual ringtests. At the first meeting of the heads of the labs next year these labs will be asked whether they are willing to help other labs by visiting them and/or inviting them to their own labs.

### **11. Preparation of a meeting of the heads of the labs**

Nils König presented a list of topics for the first meeting of the heads of the labs. It was decided to prepare a first meeting in 2008 (first half of the year) with the following topics:

- reports of the ringtest results (soil, foliar)
- discussion of the results
- discussion of specific analytical problems in the ringtests (prepared by the WG QA/QC)
- presentation of the quality paper
- presentation of the Excel sheets for control charts and ion balances
- presentation of the assistance program for labs
- presentation of the results of the evaluations about DOC in ion balances
- presentation and discussion of analytical problems proposed by the participants

The PCC of ICP Forests in Hamburg will be asked to organize the meeting (alternative: Vienna, Warsaw). It will start at 14:00 on the first day and end at 13:00 on the second day. The costs have to be paid by the participants.

(Martin Lorenz (PPC Hamburg) should be asked whether it is possible to receive money for future meetings under a COST program)

### **12. Preparation of the 3<sup>rd</sup> water ringtest**

topic was cancelled

### **13. Miscellaneous:**

- The next meeting of the WG will take place together with the Soil Expert Panel Meeting (probably in Florence, April 2008). Bruno De Vos will ask our Italian colleagues whether they can prepare the meeting for the WG. The topics of this meeting will include:

- ringtests soil (5.) and foliar (10.)
- preparation of the meeting of the heads of labs
- preparation of a list of good labs
- final discussion on the new quality check paper

Göttingen, 8.10.07

Nils König

## Attachment 1:

### Confirmed Structure of a QA/QC-paper with all suitable quality checks for labs (Eger, 2007)

	chapter	editor
0.	Introduction	Nils
1.	Use of reference material	Erwin
1.1	Reference material	Erwin
1.2	Local reference material	Erwin
1.3	Laboratory control standards	Erwin
2.	Use of control charts	Kirsti
	Use of control charts for local reference material or	Kirsti
2.1	laboratory control standards	
2.2	Use of control charts for blanks	Kirsti
3.	Check of analytical results	
3.1	Check of analytical results for water samples	Rosario
3.1.1	Ion balance	Rosario
3.1.1.1	Ion balance without DOC	Rosario
3.1.1.2	Ion balance with DOC	Rosario
3.1.1.3	Ion balance with DOC and metals	Nils
3.1.2	Conductivity check	Rosario
3.1.3	Na/Cl-ratio check	Rosario
3.1.4	N balance check	Rosario
3.1.5	Phosphorus concentration as contamination check	Nicolas
3.1.6		
3.2	Check of analytical results for soil and humus samples	Bruno
3.2.1	Plausible range checks for soil and humus material	Bruno
3.2.2	pH check	Bruno
3.2.3	C balance check	Bruno
3.2.4	C/N ratio check	Bruno
3.2.5	C/P ratio check	Bruno
3.2.6	pH/CO <sub>3</sub> check	Bruno
3.2.7	Extracted elements/total element check	Bruno
3.2.8	reactive elements/total elements check (Al, Fe)	Bruno
3.2.9	Exchangable elements/total elements check	Bruno
3.2.10	....	
3.3	Check of analytical results for plant and litterfall samples	Alfred
3.3.1	Plausible range checks for plant material	Alfred
3.3.2	....	
3.4	Analyses in duplicate	Nils
3.5	Avoidance of contamination problems	
3.5.1	Water analyses	Kirsti
3.5.2	Soil and humus analyses	Bruno
3.5.3	Foliar and litterfall analyses	Alfred
4.	Interlaboratory quality assurance	Nils
4.1	Ringtests and ringtest limits	Nils
4.1.1	Ringtests	Nils
4.1.2	Tolerable limits for ringtests	
4.1.2.1	Tolerable limits for water ringtests	Rosario
4.1.2.2	Tolerable limits for soil ringtests	Bruno
4.1.2.3	Tolerable limits for foliar ringtests	Alfred
4.2	Exchange of knowledge and experiences with other labs	Anna
4.2.1	Exchange of know how	Anna
4.2.2	Sample exchange	Nils
<u>Appendix</u>		
1.	Definitions and terminology	Erwin

2.	Excel worksheets for ion balance, conductivity check, N balance check and Na/Cl ratio check	
	Excel worksheet for check of ion balance without DOC, conductivity check, N balance check and Na/Cl ratio check	Rosario
2.1		
	Excel worksheet for check of ion balance with DOC, conductivity check, N balance check and Na/Cl ratio check	Rosario
2.2		
3.	Excel worksheet for control charts	Kirsti
	language check of the whole paper	John