

**European Union / United Nations Economic Commission for Europe
International Co-operative Programme on Assessment and Monitoring of Air Pollution Effects
on Forests**

Meeting of the Working Group QA/QC in Labs

January 13th 2009, Hamburg, Germany

Minutes (Combined FutMon and ICP-Forests Meeting)

0. Introduction

Nils König welcomed the members of the Working Group in Hamburg. He introduced Anna Kowalska as co-chairperson (proposed at the Florence meeting in 2008, to be approved at the Task Force Meeting in St. Petersburg 2009) to the participants and presented the agenda of the meeting.

1. Ring tests (water, soil, foliage, soil physics):

A proposed timetable for the ring tests has been revised. Four ringtests are planned for 2009: soil, water, foliar, and soil physics. The participation in the ring tests is mandatory for all laboratories which are analysing samples within the FutMon project. Responsibles for sample preparation and sending, data evaluation, and elaboration of the reports: A. Fürst (foliar ring test), B. de Vos and N. Cools (soil and soil physics ring tests), K. and J. Derome (sample preparation and sending water ring test), R. Mosello (evaluation and report water ring test) confirmed the preparedness. A data input module used in previous foliar ring tests has been installed and is ready to use in the forthcoming ring tests (except soil physics) as corroborated by A. Fürst. A timetable for soil physics has been proposed by FSCC representatives and accepted. Sending water samples, originally planned for February 2009 had been moved to March by necessity to ascertain full list of laboratories that will perform analyses in frame of the FutMon project. vTI will mail next week (19-24.01.2009) a request to all beneficiary NFC's to send back as soon as possible all information about their laboratories. After completing the list of prospective participants by K. Derome and A. Fürst invitation will be sent at the beginning of February. Water testing laboratories will also get a reminder on complying with QA checks. The deadline for registration has been set at the end of February. For the next foliar ring test registration will be appointed at some future date. Also decision on data submission deadline in 2010 will be taken in future time. The reports for foliar and soil ring tests are going to have the same content as previously. Data of water ring test will be elaborated according to ISO standard. All reports will contain the table with visual designation of performance of the lab for each parameter. Draft reports with the results of the 4 ring tests will be sent to the participants in September 2009 and discussed at the Meeting of the Heads of the Labs to be held in Warsaw in October 2009. (see attached the minutes of an informal meeting for the preparation of the water ring test: annex 1)

Timetable ringtest 2009 (FutMon) and meetings

Month	1	2	3	4	5	6	7	8	9	10	11	12	Jan 2010
Soil			sending samples			results from Participants			evaluation of data, draft report			final report	
Water			sending samples		results from Participants				evaluation of data, draft report			final report	

Foliage	results from Participants	evaluation of data, draft report	final report				sending samples (next ringtest)				results from Participants (next test)
Soil physics		sending samples				results from Participants		evaluation of data, draft report			
WG QA/QC	Combined Meeting Hamburg										
Heads of the Labs									Meeting Warsaw: Discussion of the 4 ringtest results (yellow)		

2. benchmark of the ring tests

Benchmarking of the ring tests has been discussed.

After the ring test each participant will receive a qualification report. The form of the report proposed by A. Fürst and N. König has been accepted to use in future ring tests.

It has been decided to qualify the results of each parameter, if 50% or more of the results for this parameter for all the samples of the ring test are within the tolerable limits (listed in the quality check paper of the WG). Missing data (mandatory parameter not analysed) is a reason for the lack of qualification. High within-lab variation and high limit of quantification for a parameter will be remarked in the qualification report (see annex 2) and in future can be incorporated in the criteria for qualification.

The proposal for a requalification procedure has been accepted. It will consist in reanalysis of the ring test samples, report to the WG QA/QC with the new results together with the original reports of the instruments and information about weight factors, dilution factors etc. and information about the reasons for the unsatisfactory results during the ring test. Alternatively: assistance program for the lab with bad ring test results is launched; then reanalysis of the ring test samples, report to the WG QA/QC with the new results together with the original reports of the instruments and information about weight factors, dilution factors etc. and information about the reasons for the bad results during the ring test. Requalification report is drawn up after positive decision of the persons in charge for the different ring tests (in case of doubt: WG QA/QC) about the report from the lab.

Stability of the samples used for reanalysis will be checked by the persons in charge for the preparation of the ring tests samples, in case of water samples also from a German (N. König) and an Italian (R. Mosello) laboratory.

3. Opening of the lab codes

After meeting of the heads of the labs in Hamburg 2008 a letter to labs was sent to ask them to release their lab ID within this group. In case they do so, they can benefit from the information present in a technical info database on laboratory equipment, instruments etc. In case they do not, they won't have access to the codes of the other laboratories. *It is intended that this information will be posted on a password protected section of the ICP Forests website and will be made available to all participating laboratories, their heads and the WG QA/QC in Laboratories only.* General reaction from the lab was positive (13 positive reactions and no reaction against this proposal). On the next Task Force Meeting in St. Petersburg in May 2009 WG QA/QC in Laboratories will inform all NFC's about decision of the heads of the laboratories.

It was decided to abstain from a password protected section of the website for the codes. Instead of that laboratories ought to confirm their decision on releasing their codes during the registration

procedure of the next ring tests by marking their agreement. List of released codes will be sent to the heads of the labs along with the report as a hardcopy.

4. helping program for labs with bad ring test results

Assistance program for lab with bad ring test results will be continued within the frame of FutMon. In the final proposal of the project 10 visits of laboratories under C1 actions are possible. Currently one laboratory requested for help. First contact questionnaire has been sent; the WG group is still awaiting the response.

Assistance to the labs can be also given through free exchange of knowledge via google group WG QA/QC in Labs, established in 2008. Laboratories will be informed about this possibility as well.

5. revision of the quality check paper to become submanual

In May 2008 a paper "Quality Assurance and Control in Laboratories - a review of possible quality checks and other forms of assistance" was published on the ICP-Forests web-page (<http://www.icp-forests.org/DocsQualLab/QualCheckMay2008.pdf>). The Quality Committee decided that this paper should be the basis of a new ICP Forests submanual "Quality Assurance and Control in Laboratories". Therefore the document needs some revision (e.g. restructuring, adding a chapter on quality indicators) to become submanual. It was decided that a small group (K. Derome, N. Cools, N. Clarke, T. Jakovljevic, P. O'Dea) under the leadership of A. Kowalska shall elaborate a proposal for the new submanual. It was decided to use actual version of the paper with some amendments as the draft field protocol for the FutMon project.

6. discussion of the quality indicators

After a short discussion about the proposed quality indicators at the EPD meeting in Rovaniemi, Finland, three quality indicators have been chosen:

1. Percentage of the results of the ring tests within tolerable limits for each ring test.
2. Percentage of the results of the ring tests of repeatability below 10% (not for water ring tests).
3. Mean percentage of parameters for which laboratories use control charts.

First two of them can be inferred from results of the ring tests. Third must be obtained from laboratories (as e.g. an answer submitted with the ring test results or from the quality report forms, see topic 9).

7. Use of the quality checks in practice

Numerous quality checks for integrity of data are listed in the document "Quality Assurance and Control in Laboratories - a review of possible quality checks and other forms of assistance". Use of them is strongly recommended to labs to assure quality of data. The laboratories are in charge of the data quality.

A link between data and their quality must be maintained in the database.

The possibility of integrating of quality checks into data reports has been discussed. The information on data if passed or not ion balance might be integrated in a new form (quality form); that needs to be discussed with the database manager (see topic 9).

The reason of missing data, as e.g. small sample volume, contamination, may be also encoded and combined into data reports.

8. Detection/Quantification limits

N.König presented the method of assessment the LOQ widely used in German laboratories, whereas the presentation by G. Tartari will possibly be shown at the next meeting of the heads of the labs.

It was decided to report and use only the quantification limit, not the detection limit.

Data below quantification limit are marked in database by "-1" and it will not be changed, but the value of method/matrix limit of quantification must be linked to these data.

Oliver Granke proposed to remove the section on missing data and values below LOQ from deposition, foliar and soil submanuals and gather them in the quality submanual with definitions and description of how to report these data.

9. Discussion about the data submission formats with vTI (O. Granke)

Oliver Granke from vTI presented current construction of the database records. Additional parameters proposed by WG QA/QC can be easily integrated in the database, provided that existing units are not changed and number of digits is invariable with floating decimal point.

New quality forms will be constructed, separately for deposition, foliar, soil and soil solution data.

The following information/quality parameters were proposed to be included in the new forms:

- beneficiary/country code
- year
- plot No
- LOQ for each parameter
- detection method (coded like in ring test reports) for each parameter,
- ring test No
- lab code
- ranking of lab in the ring test (% of results within tolerable limits)
- mean and standard deviation for each parameter from control charts

Data to the forms come from labs internal quality control and from ring test results; both types of information are available for the labs, therefore labs will fill in the new forms.

Data submission forms should combine both: mandatory and optional parameters.

Database has not got any given data completeness limits, if necessary, completeness of results can be marked in extra added column in %.

O. Granke will elaborate the new forms and will circulate them among WG members for comments.

Forms will be submitted to the Task Force Meeting in May 2009 for approval, then presented in October 2009 at the meeting of the heads of the labs.

A new working group "data management" (with data managers of the beneficiaries) was proposed.

The WG will constitute if decision is reached at the meeting of data managers in September 2009.

10. Second meeting of the heads of the labs (Warsaw, October 2009)

Anna Kowalska proposed 12.-13. or 19.-20. October for the meeting.

At the first meeting of the heads of the labs in Hamburg it was decided to ask participants for presentations about the following analytical problems:

- Problems with digestion methods for plant material (microwave and other systems)
- Problems with Aqua Regia digestion for soils (microwave and other systems)
- problems with ICP and AAS measurements in extracts and digested solutions
- problems with DOC and TN measurements in water samples
- comparison of results from different instruments

Results of the ring tests as well as consequences of non-qualified results, data submission, presentation of the google group WG QA/QC in Labs, and some analytical problems will be raised at the sessions.

The preliminary topics for the agenda has been accepted as below:

Topic	Presentation(s)	Time
Report of the ringtest results (soil)	N.Cools	1:15
Report of the ringtest results (foliar)	A.Fuerst	1:00
Report of the ringtest results (water)	R.Mosello	1:15
Report of the ringtest results (soil physics)	N.Cools	0:45
Information about qualification/requalification reports	N.Koenig	0:20
Assistance program for labs	N.Koenig	0:10

Data submission: new form for quality information	O.Granke/A.Kowalska	0:30
Information about the opening of the lab code	A.Fuerst	0:05
Presentation and discussion of analytical problems proposed by the participants (see list above)		2:00
Detection/quantification limits – determination and	G.Tartari	0:25
Problems with digestion methods for plant material	A.Fuerst	0:20
Problems with Aqua Regia digestion for soils	Participants	1:00
Google Group/web page	N.Koenig	0:30
FSCC reference sample	N.Cools	0:20
Other business		0:30

8-10 weeks before the meeting a letter will be sent to the possible participants by N. König with a request for presentations about analytical problems and proposals of other topics.

11. Discussion about the maximum sample storage period

N. Cools outlined the requirements of ISO standard 18512 related to current practice in 20 countries/ 25 institutions. Most of the countries store samples for long-term monitoring, with a limited experience of change of properties with time. Minimum-maximum requirements for storing conditions should be set, controlled and reported (DAR-Q).

N. Koenig presented results of a few years tests of standard materials. Some measured values had been changing over the observed period. Some of the elements are more than other vulnerable to changes (e.g. exchangeable Mn, Fe and H, pH).

In foliar material changes although appear, but they are less frequent than in soils.

Chemical composition of water is highly susceptible to alteration, especially at pH >4,5. Some information on the storage of water samples can be found in Analytical Info Sheets at: http://www.icp-forests.org/WGqual_lab.htm.

Changes of chemistry of samples in time are not easy, if at all possible to avoid, therefore storage conditions have to be controlled and documented.

12. Miscellaneous

a. A. Fuerst proposed new tolerable limits for low concentration (e.g. non-foliar litter, branches), basing on the results from last ring test. New limits are broader than for normal, higher concentration in foliage.

Element concentrations below	Tolerable deviation from the mean (+%)	for
Sulphur	20	0.5 mg/g
Phosphorous	15	0.5 mg/g
Magnesium	15	0.5 mg/g
Zinc	20	20 µg/g
Manganese	20	20 µg/g
Iron	30	20 µg/g
Lead	40	0.5 µg/g

Boron	30	5 µg/g
Nitrogen	15	5 mg/g
Potassium	15	1 mg/g

b. Last foliage ring test (11th) first results will be published on the internet web-site on Monday, 19 Jan.

First evaluation revealed some analytical problems: results of C, Mg, Ca got worse and in sample containing branches. A digestion method problem might have occurred, as indicate the results of Fe and Cu.

c. The date of the next WG meeting will be decided bei vTI (combined meeting).

20.01.2009

A.Kowalska / N. König

annex 1:

Informal meeting Derome Kirsti, Nils Koenig, Rosario Mosello

Hamburg, 14 January 2009

Intercomparison exercises, with attention to the atmospheric deposition and soil water exercise

The intercomparison exercise dealing with atmospheric deposition and soil water, in the framework of the FutMon project, will be carried out following as strictly as possible the ISO/IEC rules, e.g.

IUPAC Technical report 1/2006

The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories

ILAC – G13:2007

ILAC guidelines for the Competence of Providers of Proficiency Testing Schemes

ISO/IEC CD 17043:2008

Conformity Assessment – General requirements for proficiency testing.

The most important differences with the two previous intercomparisons (WRT 2002 and 2005) are:

- 1) the need to previously detail to the participants all the steps and methods involved in the intercomparison and
- 2) the lack of screening on the digitation and units of the submitted results (particular relevance for nitrate and alkalinity, often mailed with units different from those required).

Samples will be prepared from METLA (Derome), which will perform **preliminary analyses** to assure the needed and agreed range of concentrations and the **homogeneity test** among bottles.

Stability of the solutions will be tested for a period of six months from METLA, CNR ISE and Nordwestdeutsche Forstliche Versuchsanstalt. ICP Forests laboratories not directly participating in the FutMon project will be as well involved in the intercomparison.

With the help of vTI Hamburg a list of all FutMon labs will be collected until 6th Feb. 09 and then combined with the list of ICP Forests labs and others.

From this complete list Alfred Fürst will prepare 3 lists of the labs for the soil, water and plant ring test with the lab-codes and passwords for the registration. He will send these 3 lists to the persons in charge for the 3 ring tests.

Four documents will prepare and accompany the intercomparison exercises:

- 1) General invitation letter dealing with the three intercomparisons (soil, plant and water), written by Nils König (and Alfred Fürst).
- 2) Detailed letters specific for each of the three intercomparisons from the three persons in charge for water, soil and leaves tests. In the letter information must be given about lab codes and passwords for the pre-registration, using the software prepared by Fürst.
- 3) Detailed letters to the registrated laboratories specific for each of the three intercomparisons from the three persons in charge for water, soil and plant tests, giving all the technical information on the process and methods used and warnings for the correct submission of data (e.g. attention to the units, any correction of the results forbidden, how to mail the results, etc). Minute to Alfred, Nils, Kirsti, John, Bruno, Nathalie.
- 4) Accompanying letter of each type of intercomparison, giving details on the range of expected concentration, timing for the analyses and other practical details concerning the analyses and the mailing of data. (Prepared by the person/institute in charge of the preparation and mailing of the results).

Copies of these letters should be available both in the Fürst's web page and in the ICP Forests QA/QC web page, where a specific space for the intercomparisons should be present, to disseminate the different documents, including expected results, draft and final reports, etc. (König).

The persons/labs in charge of the data elaboration will prepare, together with the summary of results and the draft report, a "Qualification report" for each laboratory, where a summary of the

performances will be published. The report will document, for each parameter, if analysed or not, if passed to the test, and notes (see annex). Not analysed mandatory variables will be considered as “not passed”. This qualification report will be mailed as well to the National Focal Centres.

Laboratories can submit to the person in charge of the intercomparison exercise a “Re-qualification report”, where he documents the reason of the error(s) and the improvements done to make reliable the considered analysis. If accepted the re-qualification will be documented and mailed to the laboratory and NFC. For atmospheric deposition/soil water this will be done by CNR-ISE with the help of METLA and Nordwestdeutsche Forstliche Versuchsanstalt.

Draft report of the results will be circulated before the meeting of the head of laboratories in Warsaw (mid October), results will be presented and discussed in the meeting, and the points emerging from the discussion will be considered for the preparation of the final report.

annex 2:

Qualification Report (example)

11th Needle/Leaf Interlaboratory Comparison Test 2007/2008

Head of the lab

Institut

Street

Town

country


Labcode: 34

Parameter	analysed	passed	not passed	Remarks	Requalification	
					Report to FFCC	Repetition report + protocol
N	X	X				
S	X		X		11.02.2009	
P						
Ca						
Mg	X	X		high laboratory standard deviation (Si)		
K	X		X			30.03.2009
Zn	X	X		high determination limit		
Mn						
Fe						
Cu						
Pb	X	X				
B	X	X				
Cd						
C						

passed: 50% of the results per element are within the tolerable limits

not passed: more than 50% of the results per element are outside of the tolerable limits

22.01.2009



Alfred Fürst
Forest Foliar Co-ordinating Centre