Forest Soil Co-ordinating Centre (FSCC)

Pan European Programme on the Intensive Monitoring of Forest Ecosystems (UN/ECE ICP Forests - European Commission)

Wetenschappelijke Instelling van de Vlaamse Gemeenschap
Instituut voor Bosbouw en Wildbeheer

Quality assurance & Quality control in forest soil analysis: An interlaboratory comparison

Cools N., Delanote V., Scheldeman X., Quataert P., De Vos B., Roskams P.

Forest Soil Co-ordinating Centre - Institute for Forestry and Game Management
Gaverstraat 4 - B-9500 Geraardsbergen - BELGIUM
E-mail: FSCC@vlaanderen.be
Tel. + 32 54 43 61 59 Fax + 32 54 43 61 60

Introduction

A frequently faced problem in transnational monitoring programmes is finding a balance between **international reference methods**, improving spatial comparability, and **national analysis methods** that favour, by their use and testing over many years, temporal comparability. Fifty-two soil laboratories from 27 European countries, participated in an interlaboratory comparison, organised in 2002 by the Forest Soil Co-ordinating Centre (FSCC), one of the four thematic centres of the **Pan European Programme on Assessment and Monitoring of Air Pollution Effects on Forest Ecosystems (EC – UN/ECE ICP Forests)**. The ring test was part of an overall quality assurance programme to enhance the quality of the European Forest Soil Database in the perspective of a new Pan European forest soil survey.

Plate 1: sampling location of sample B

Materials and methods

Plate 2: Sampling of sample C

A total of 48 soil parameters were analysed on three forest soil samples. While sample A and B were mineral samples, sample C was an organic layer sample. The laboratories were requested to follow the **reference methods for soil analysis**, as outlined in the ICP Forest Manual, of which the part on sampling and analysis of soils has been updated recently (FSCC, 2003). By completing a questionnaire, each laboratory provided information on their analysis method, equipment, experience and quality control procedures.
Two aspects of data consistency have statistically been judged: the **inter-laboratory variability** (based on the Mandel's h statistic) and the **intra-laboratory variability** (based on the Mandel's k statistic).
In view of **follow-up** to improve the inter-laboratory variability, an exploratory analysis was conducted to retrieve any relationship between the variability of the ring test results and information provided through the questionnaire (as use of reference methods, training of personnel, experience, etc.) by both univariate (boxplots, ANOVA and Kruskal-Wallis tests) and multivariate analysis (multiple stepwise regression).

Results and discussion

The followed procedure studied the occurrence of **outliers** (99% chance that reported analysis is outside acceptable range) and **stragglers** (95% chance outside acceptable range). There were **more** laboratories with outlying results concerning the intra-laboratory variance compared to the inter-laboratory variance (Table 1). Laboratories **reporting more than 20%** outliers or stragglers need to improve strongly and need help through a follow-up programme.
Sample A, which was a chemically poor mineral topsoil sample, caused **more difficulties** in analysing than sample B and C (see Figure 1, 2 and 3 and Table 2).

Table 1: N° of laboratories reporting more than 20% outliers or stragglers

Sample	N° of laboratories			
	Total N° laboratories	Excluded based on Mandel's:		
		h	k	h and k
A	51	4	11	4
B	51	1	2	1
C	52	2	6	2

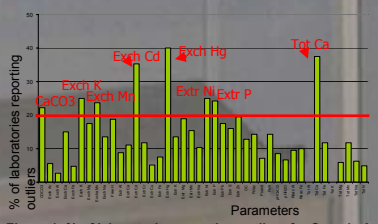

Figure 1: % of laboratories reporting outliers for Sample A

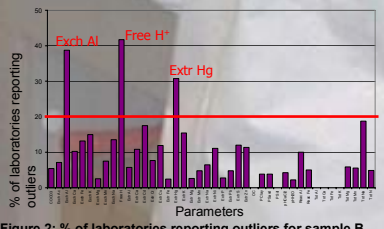

Figure 2: % of laboratories reporting outliers for sample B

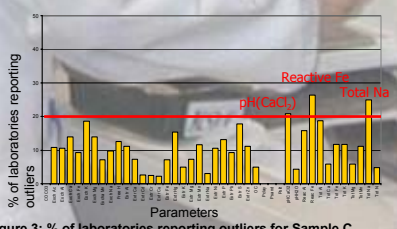

Figure 3: % of laboratories reporting outliers for Sample C

Table 2:CVs among the laboratories per parameter group

Parameter group	Sample A	Sample B	Sample C	Mean
pH	4.1	2.3	4.0	2.8
Particle size	41	47	NA	53
Carbonates	374	38	NA	206
Organic carbon	11	33	9.7	18
Total N	36	6.9	6.3	17
Exchangeable elements	72	105	37	71
Extractable elements	87	27	29	47
Total elements	32	19	10	21
Reactive Al and Fe	42	40	51	44
Average per sample	**70**	**43**	**26**	

The parameters could be divided into **3 groups** according to the **difficulty to analyse** and faced problems. pH, total N and OC were relatively easy to analyse; total elements and elements extracted by aqua regia were more difficult and elements extracted by acid oxalate, particle size distribution, exchangeable elements and total carbonate content showed very high coefficients of variation (CV).
Because the design of this ring test was not powerful enough to conduct a causal study (unbalanced observations and interaction effects between exploratory variables), **no consistent relationship** between the inter- or intra-laboratory variability and the **information** provided through the **questionnaire** could be established.

Conclusions and recommendations

The variability between and within the laboratories was large and **need improvement** in view of a next Pan European forest soil survey. A **follow-up** ring test using the same statistical procedures is highly recommended and is planned in the near future.

Reference

FSCC. 2003. Manual on methods and criteria for harmonized sampling, assessment, monitoring and analysis of the effects of air pollution on forests. Part IIIa. Sampling and Analysis of Soil. UN/ECE Convention on the Long-Range Transboundary air Pollution, International Co-operative Programme on Assessment and Monitoring of Air Pollution Effects on Forests and the European Union Scheme on the Protection of Forests against Atmospheric Pollution