

**23<sup>th</sup> Task Force Meeting of ICP Forests  
Zvolen, Slovak Republic, 12-16 May 2007**

**Importance of  
quality assurance and quality control  
for  
good and comparable analytical results  
in ICP Forests programs**

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for the Working Group on QA/QC in Labs**

Importance of **quality assurance and quality control** for **good and comparable analytical results** in ICP Forest programs

## what we need:

1. good „equipment“ in the laboratory
- ② quality control program within each laboratory

**The laboratories and the NFC's are responsible for the quality of the analytical data!**

3. quality and plausibility checks before importing of analytical data into national and European databases
- ④ quality control between the different European laboratories and exchange of analytical knowledge

Importance of **quality assurance and quality control** for **good and comparable analytical results** in ICP Forest programs

## **1. good „equipment“ of the lab:**

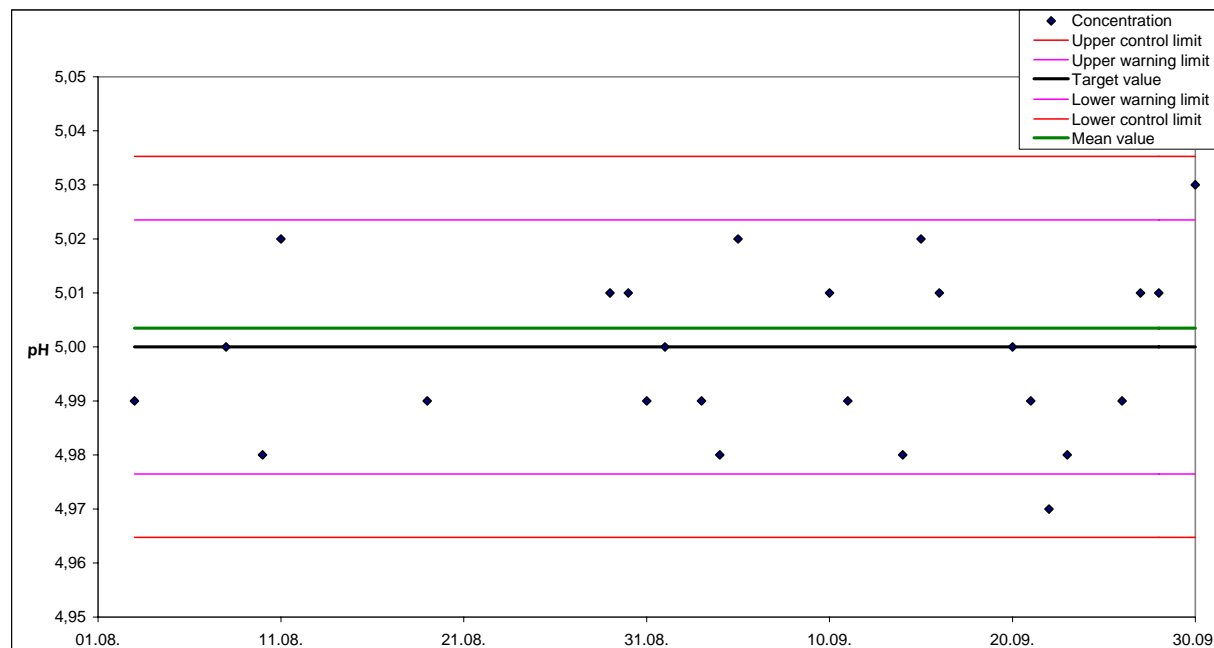
- a. good technicians with experience in the European reference methods
- b. good instruments
- c. good method descriptions (for sample preparation, digestion/extraction methods and element determination)

Importance of **quality assurance and quality control** for **good and comparable analytical results** in ICP Forest programs

## 2. quality control program within each laboratory:

a. regular use of standard material for method control and use of control charts

Institute		X-CONTROL CHART			METHOD: ROLVaxx		CONTROL SAMPLE:	
Laboratory		PERIOD: 03.08.04 - 30.09.04			INSTRUMENT: MagicChem		Reagecon 5.00	
Analyte	Target value (Tv)	Warning limits (Tv ± 2STD)		Control limits (Tv ± 3STD)		Mean value □ □	Standard deviation (STD)	
pH	5,000	Upper 5,024	Lower 4,976	Upper 5,035	Lower 4,965	5,003	0,012	



[as a help for the labs: excel file with a program for control charts](#)

**Importance of quality assurance and quality control for good and comparable analytical results in ICP Forest programs**

**Results from the questionnaires  
of the 3<sup>rd</sup> and 4<sup>th</sup> FSCC soil ringtests:**

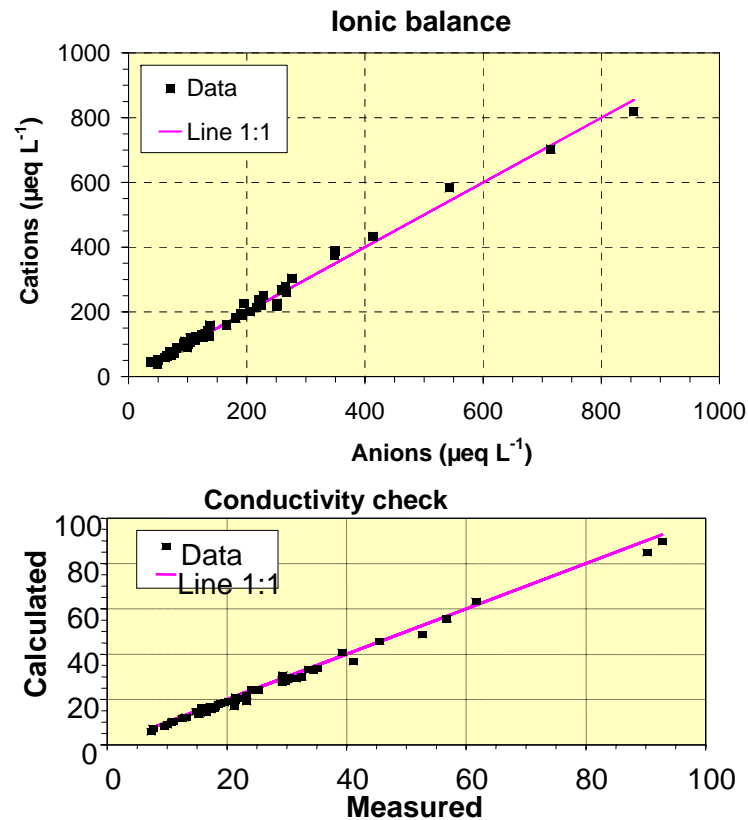
Questionnaire	3 <sup>rd</sup> FSCC RT (2002 - 2003)	4 <sup>th</sup> FSCC RT (2005 - 2006)
Use of reference material	77 %	86 %
Use of control charts	50 %	65 %
Use of calibration standards	73 %	63 %

**=> reference material, control charts and calibration standards should be used by 100 % of the labs !**

# Importance of **quality assurance and quality control** for **good and comparable analytical results** in ICP Forest programs

## 2. quality control program within each laboratory:

b. use of all important plausibility and quality checks **directly after analyses**



Microsoft Excel - EPD\_analytical\_data\_validation

as a help for the labs:  
excel file with a  
program for  
calculation of ion  
balance,  
conductivity check,  
Nitrogen balance,  
Na/Cl-ratio

ICP Forests																		
Sampling station: Example																		
Please fill in only green cells																		
Starting day	End day	mm	pH	$\gamma_{25^\circ\text{C}}$	Ca	Mg	Na	K	N-NH <sub>4</sub>	S-SO <sub>4</sub>	N-NO <sub>3</sub>	Cl	T. Alk.	PO <sub>4</sub>	TN	DOC	O	
			$\mu\text{S cm}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\mu\text{eq L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$	$\text{mg L}^{-1}$
28/12/2004	04/01/2005	27,2	5,89	4,8	1,12	0,5	3,50	0,12	0,255	0,82	0,232	9,70	8	0,008	0,43	-0,		
04/01/2005	11/01/2005	119,7	6,65	29,7	1,29	0,39	2,53	0,22	0,106	0,49	0,128	5,00	70	0,003	0,41	0,		
11/01/2005	18/01/2005	70,9	6,18	10,2	0,22	0,2	0,11	0,22	0,12	0,31	0,12	3,3	10	0,000	0,61	0,		
18/01/2005	25/01/2005	48,0	6,08	1,6	0,2	0,1	0,4	0,1	0,1	0,1	0,1	1,7	15	0,004	0,29	0,		
25/01/2005	01/02/2005	104,5	4,94	15,7	0,22	0,15	1,33	0,14	0,055	0,40	0,176	2,24	0	0,000	0,46	0,		
01/02/2005	08/02/2005	28,6	6,21	1,5	0,5	0,19	1,27	0,20	0,100	0,40	0,235	1,80	20	0,000	0,57	0,		
08/02/2005	15/02/2005	29,7	5,45	3,4	0,1	0,1	0,1	0,18	0,470	1,13	0,540	4,10	0	0,003	1,26	0,		
15/02/2005	22/02/2005	92,7	5,58	31,6	0,47	0,47	3,60	0,23	0,130	0,64	0,170	5,97	0	0,001	0,44	0,		
22/02/2005	01/03/2005	10,2	6,44	90,2	4,29	1,24	7,00	0,71	0,810	2,70	1,05	12,80	11	0,015	2,21	0,		
01/03/2005	08/03/2005	45,7	5,99	6	0,4	0,1	0,4	0,1	0,1	0,1	0,1	6	6	0,004	0,37	0,		
08/03/2005	15/03/2005	56,4	5,98	18,0	0,56	0,16	1,35	0,18	0,320	0,65	0,300	1,92	15	0,001	0,75	0,		
15/03/2005	22/03/2005	55,5	4,66	4,1	0,34	0,47	2,87	0,22	0,270	0,33	0,370	6,12	0	0,001	0,83	0,		
22/03/2005	29/03/2005	68,1	5,67	2,1	1,2	0,4	0,2	0,3	0,1	0,1	0,1	4,3	11	0,002	0,42	0,		
29/03/2005	05/04/2005	148,2	5,95	17,9	0,70	0,18	3,34	0,10	0,150	0,67	0,105	2,37	10	0,002	0,29	0,		
05/04/2005	12/04/2005	26,8	4,74	20,3	0,44	0,13	0,45	0,13	0,460	0,81	0,456	0,66	0	0,000	0,93	0,		
12/04/2005	19/04/2005	106,2	4,98	1,5	0,1	0,06	0,2	0,2	0,4	0,76	0,284	0,30	0	0,001	0,85	0,		
19/04/2005	26/04/2005	53,4	5,23	1,6	0,37	0,19	1,56	0,34	0,110	0,56	0,402	2,60	0	0,003	0,62	0,		
26/04/2005	03/05/2005	21,9	4,54	33,7	1,49	0,21	0,98	0,27	0,340	1,40	0,770	1,73	0	0,002	1,34	0,		
03/05/2005	10/05/2005	16,3	6,36	20,3	1,43	0,16	0,79	0,21	0,490	0,83	0,482	1,25	16	0,001	1,15	0,		
10/05/2005	17/05/2005	22,7	5,64	35,1	1,29	0,43	3,37	0,48	0,100	0,97	0,380	6,05	0	0,001	0,54	0,		
17/05/2005	24/05/2005	65,6	6,29	21,9	0,75	0,24	1,90	0,14	0,260	0,46	0,190	3,28	1	0,006	0,27	0,		

## Importance of **quality assurance and quality control** for **good and comparable analytical results** in ICP Forest programs

### b. use of all important plausibility and quality checks

#### b.1. for water samples:

- completeness of analyses (all parameters)
- ion balance without DOC (only for bulk or filtered)
- ion balance with DOC for throughfall, stream water
- conductivity check (comparison between samples)
- Na/Cl ratio check (not for soil water)
- Nitrogen balance ( $\text{NH}_4 + \text{NO}_3 < \text{N}_{\text{tot}}$ )

#### b.2. for soil samples:

- pH check ( $\text{pH}(\text{H}_2\text{O}) > \text{pH}(\text{CaCl}_2)$ )
- Carbon balance ( $\text{C}_{\text{CO}_3} + \text{C}_{\text{org}} < \text{C}_{\text{tot}}$ )
- Plausible range checks for individual parameters
- comparison between overlying layers
- Checks based on simple relationships between parameters
- Exchangeable cation 'balances' (CEC, B<sub>+</sub>)

#### b.3. for plant samples:

- Plausible range checks for individual parameters

The working group  
on QA/QC  
will prepare a list  
containing  
all the suitable quality checks,  
including control charts  
and plausible ranges  
for soil and plant analyses,  
and send this list  
to all the laboratories  
and NFC`s

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### **3. quality and plausibility checks before importing of analytical data into national and European databases**

- a. data checking program (check of plausible ranges, balance checks and so on) at the European data bases
- b. Information about errors associated to measurements (CV %) evaluated from control charts
- c. Information about data quality evaluated from ringtests results of the labs
- c. regulation for results under detection/quantification limit



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# 4. quality control between the different European laboratories and exchange of analytical knowledge

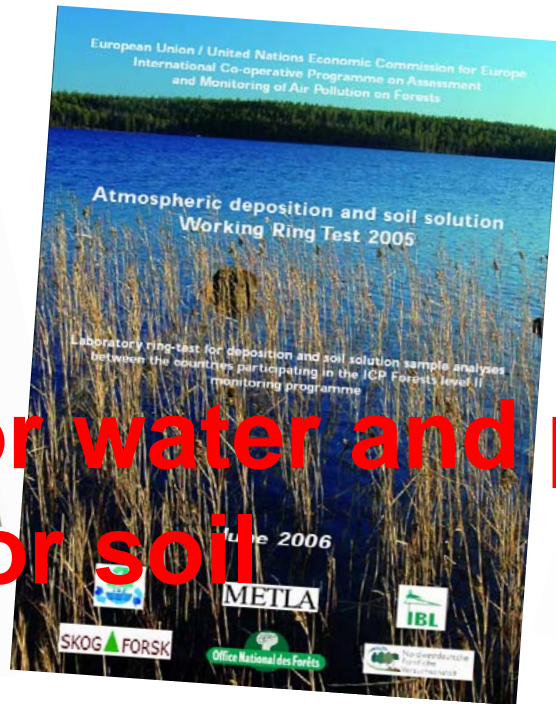
a. regularly ringtests for water, soil and plant material

CONVENTION ON LONG-RANGE TRANSBOUNDARY AIR POLLUTION  
INTERNATIONAL CO-OPERATIVE PROGRAMME ON ASSESSMENT AND  
MONITORING OF AIR POLLUTION EFFECTS ON FORESTS  
and  
EUROPEAN UNION SCHEME  
ON THE PROTECTION OF FORESTS AGAINST ATMOSPHERIC POLLUTION  
European Commission

United Nations  
Economic Commission  
for Europe

9<sup>th</sup> Needle/Leaf Interlaboratory  
Comparison Test 2006/2007

**rhythm:**  
each year for water and plant,  
all 2 years for soil



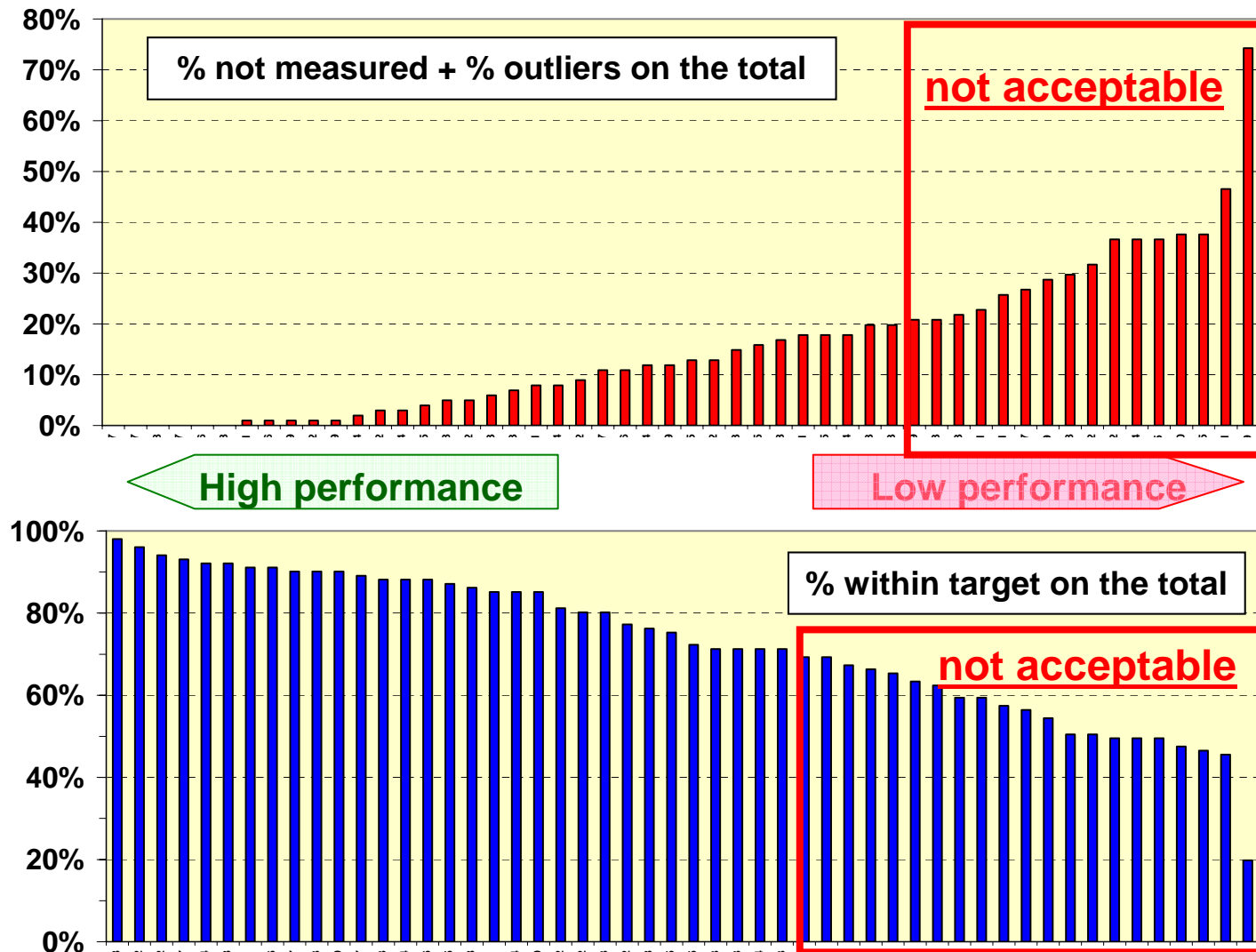
CONVENTION ON LONG-RANGE TRANSBOUNDARY AIR POLLUTION  
INTERNATIONAL CO-OPERATIVE PROGRAMME ON ASSESSMENT AND MONITORING  
OF AIR POLLUTION EFFECTS ON FORESTS  
AND  
THE FOREST FOCUS REGULATION EEC 2152/2003 OF THE EUROPEAN PARLIAMENT  
AND OF THE COUNCIL CONCERNING MONITORING OF FORESTS AND  
ENVIRONMENTAL INTERACTIONS IN THE COMMUNITY  
UNITED NATIONS  
ECONOMIC COMMISSION  
FOR EUROPE  
EUROPEAN COMMISSION  
FLEMISH COMMUNITY  
FOREST AND GREEN DIVISION

Quality Assurance and Quality Control in Forest Soil Analysis:  
4<sup>th</sup> FSCC Interlaboratory Comparison

N. Coops, P. Verschelde, P. Quatert, J. Mikkelson and R. De Vos  
2006  
INBO.R.2006.6  
FOREST SOIL CO-ORDINATING CENTRE  
RESEARCH INSTITUTE FOR NATURE AND FOREST  
GAVEWEG 14  
B-9900, GEMARSDEREN, BELGIUM  
inbo  
Institute for Nature and Forest

# Importance of **quality assurance and quality control** for **good and comparable analytical results** in ICP Forest programs

## ringtest results: 2<sup>nd</sup> water ringtest



**Importance of quality assurance and quality control for good and comparable analytical results in ICP Forest programs**

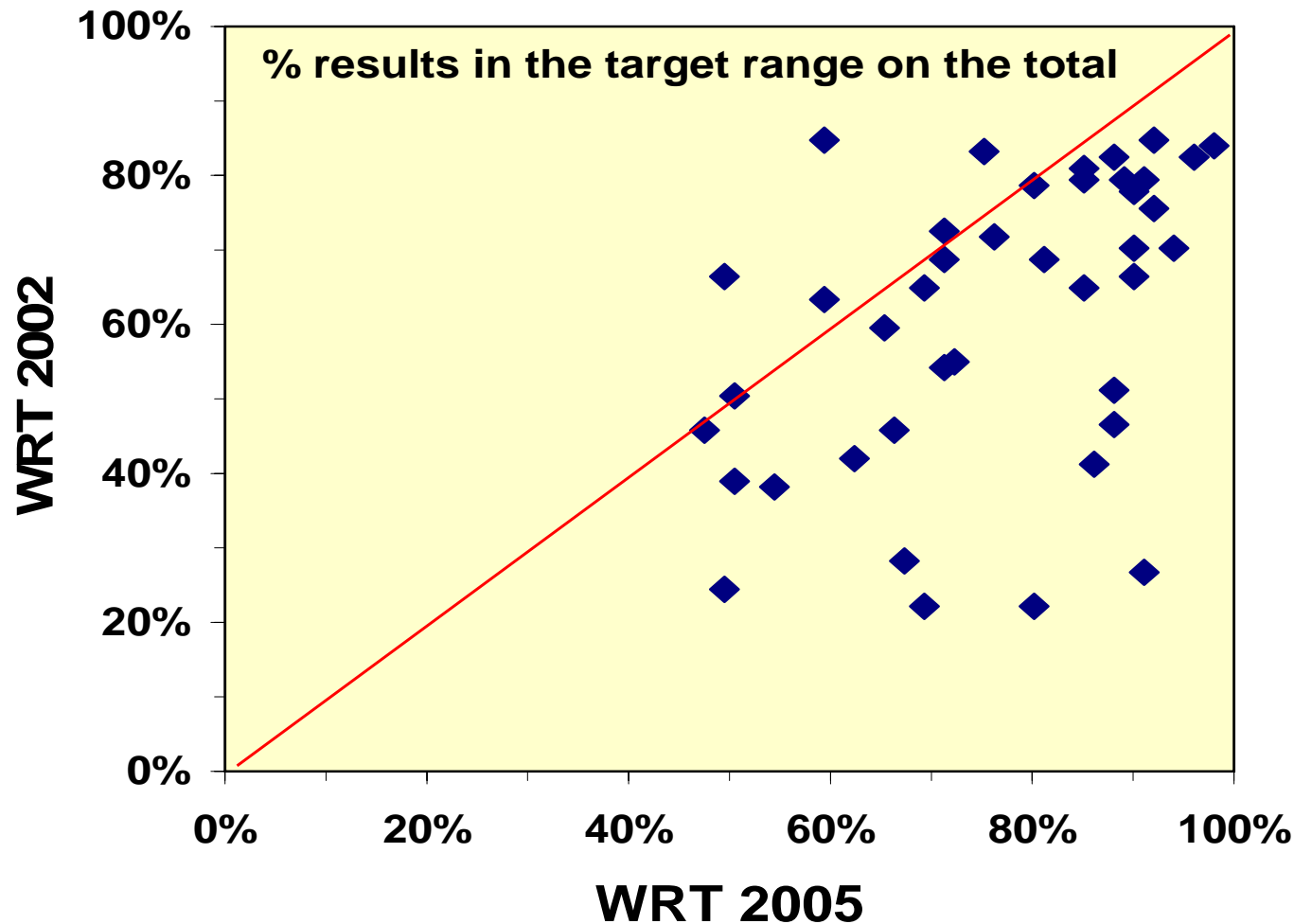
**ringtest results:  
comparison between 2., 3. and 4. soil ringtest**

Not acceptable

CV (%)	2 <sup>nd</sup> FSCC RT	3 <sup>rd</sup> FSCC RT	4 <sup>th</sup> FSCC RT
Group 1: Particle size distribution		53	37
Group 2: pH	3.25	3.5	3.1
Group 3: Carbonate content		206	129
Group 4: Organic carbon	41.5	18	13
Group 5: Total N	25	17	27
Group 6: Exchangeable cations	52	71	54
Group 7: Aqua regia extractable elements	35	47	33

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ringtest results:  
comparison between 1<sup>st</sup> and 2<sup>nd</sup> water ringtest



**More than  
85 % of  
the labs  
had better  
results in  
the 2<sup>nd</sup>  
ringtest !**

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#### **4. quality control between the different European laboratories and exchange of analytical knowledge**

b. **regularly meetings of the heads of the labs**

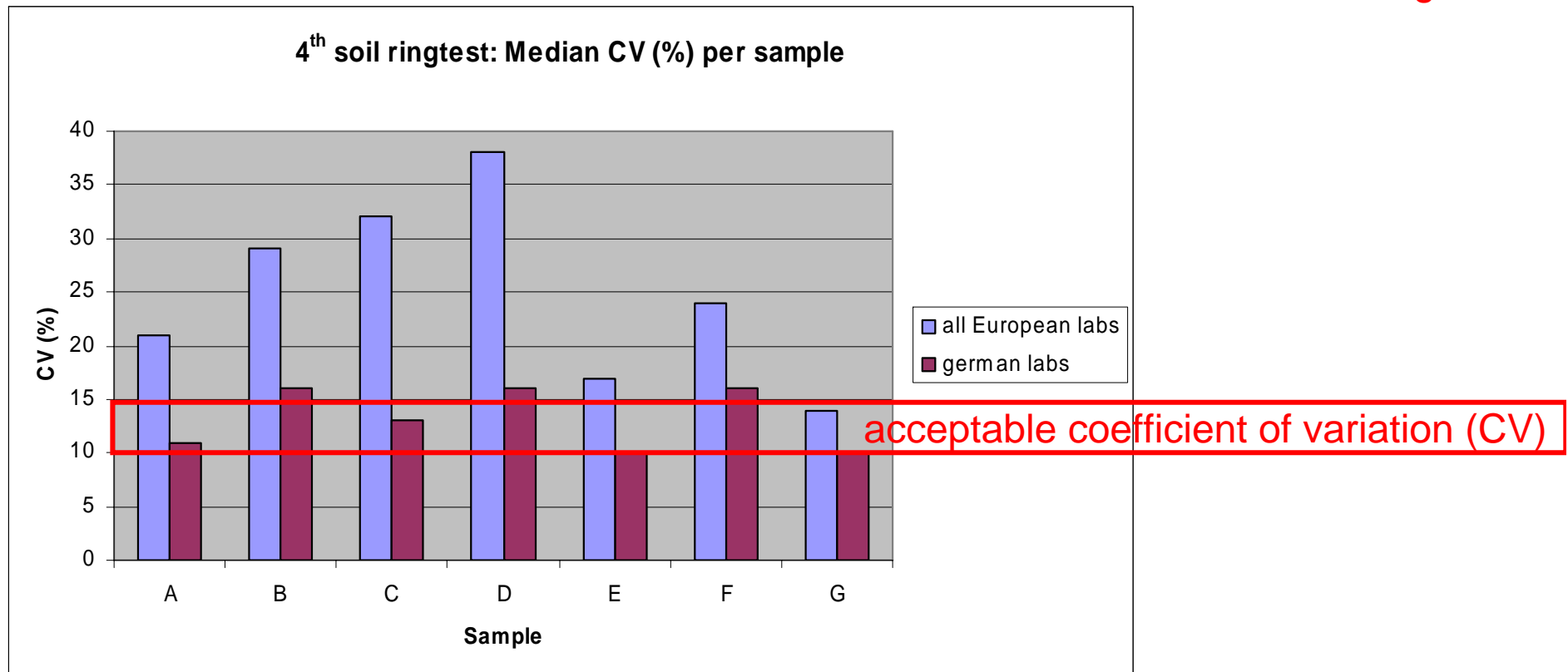
- discussion of the ringtests results
- discussion about unsuitable methods and analytical problems identified on the basis of the ringtest results
- discussion of analytical questions in the different manuals

**new!**

# Importance of **quality assurance and quality control** for **good and comparable analytical results** in ICP Forest programs

12 years ago the German government decided to install a **working group** of the **leaders of the German labs** for forestry analyses (16 labs). The **aim** of the group was to **harmonize the analytical methods** between the labs and to **organize regularly ringtests** for quality control.

This has led to **better and comparable results** and shows the **importance of the exchange of analytical knowledge** between the heads of the labs and of **regularly ringtests!**



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## Conclusions:

### what to do:

1. More information about the importance of quality control programs within each laboratory !
2. More „pressure“ to the labs (by the NFC´s) to use quality checks, control charts, reference standards !  
**The laboratories and the NFC´s are responsible for the quality of the analytical data!**
3. Ringtests carried out each year (every 2 years for soil) for intercomparable analytical data
4. Discussion about restrictions for labs/countries with unsuitable ringtest results?

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## Conclusions:

### what to do (continued):

5. Changing of the composition of the working group  
in labs:  
representatives of all the laboratories performing  
chemical analyses (soil, water, sediment, air, litter,  
Foliar and Litter) and at least one representative  
group:

Therefore I ask this Task Force Meeting  
for a new mandate  
for the WG on QA/QC in Labs!

heads of the laboratories