

Results of the 10th Needle/Leaf Interlaboratory Comparison Test and a preview on future Tests

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Austrian Federal Research and Training Centre for Forests

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**CONVENTION ON LONG-RANGE TRANSBOUNDARY AIR POLLUTION
INTERNATIONAL CO-OPERATIVE PROGRAMME ON ASSESSMENT AND
MONITORING OF AIR POLLUTION EFFECTS ON FORESTS**

United Nations
Economic Commission
for Europe

**10th Needle/Leaf Interlaboratory
Comparison Test 2007/2008**



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<http://bfw.ac.at/600/ffcc/10thtest.pdf>



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Timetable

- 2007-04: Informing the participating labs
- 2007-07-02: **Registration** of the participants via internet
- 2007-07: Submission of the four ring test samples
- 2007-10 till 2007-12: Data submission of the labs
- 2007-12-31: **Deadline of data submission**
- 2008-01-03 Evaluation according to DIN 38402/42
- 2008-02-06: First feedback from **level II labs** with bad results
- 2008-02-07: Information about results and problematic labs where sent to PCC, QA/QC-Group, Members of EP-Foliage&Litterfall
- 2008-02: **Final Report**



10th Needle/Leaf Interlaboratory Comparison Test

- 54 Laboratories / 29 Countries / 4 Samples
- Lower tolerable limits for S, P, Ca, Mg and K (Madrid 2007)
- New instrumentation questionnaire
- Fast feedback from „Level II“ - laboratories was possible; a summary was include in the report (9 „problematic“ Level-II labs; 7 of them gave a first feedback within one month!!)
- 7 labs had problems to store results in the correct units....! (especially for Cd, C)
- One lab had problems with data submission (Java Script was not activated)



Element	Tolerable deviation from mean in %	Limits fixed in the Expert Panel-Foliar (Litterfall) Meetings
N	90-110	6 th Meeting - Bonn 1999
S	85-115	10 th Meeting - Madrid 2007
P	90-110	10 th Meeting - Madrid 2007
Ca	90-110	10 th Meeting - Madrid 2007
Mg	90-110	10 th Meeting - Madrid 2007
K	90-110	10 th Meeting - Madrid 2007
Zn	85-115	8 th Meeting - Prague 2003
Mn	85-115	8 th Meeting - Prague 2003
Fe	80-120	6 th Meeting - Bonn 1999
Cu	80-120	8 th Meeting - Prague 2003
Pb	70-130	6 th Meeting - Bonn 1999
Cd	70-130	6 th Meeting - Bonn 1999
B	80-120	6 th Meeting - Bonn 1999
C	95-105	6 th Meeting - Bonn 1999



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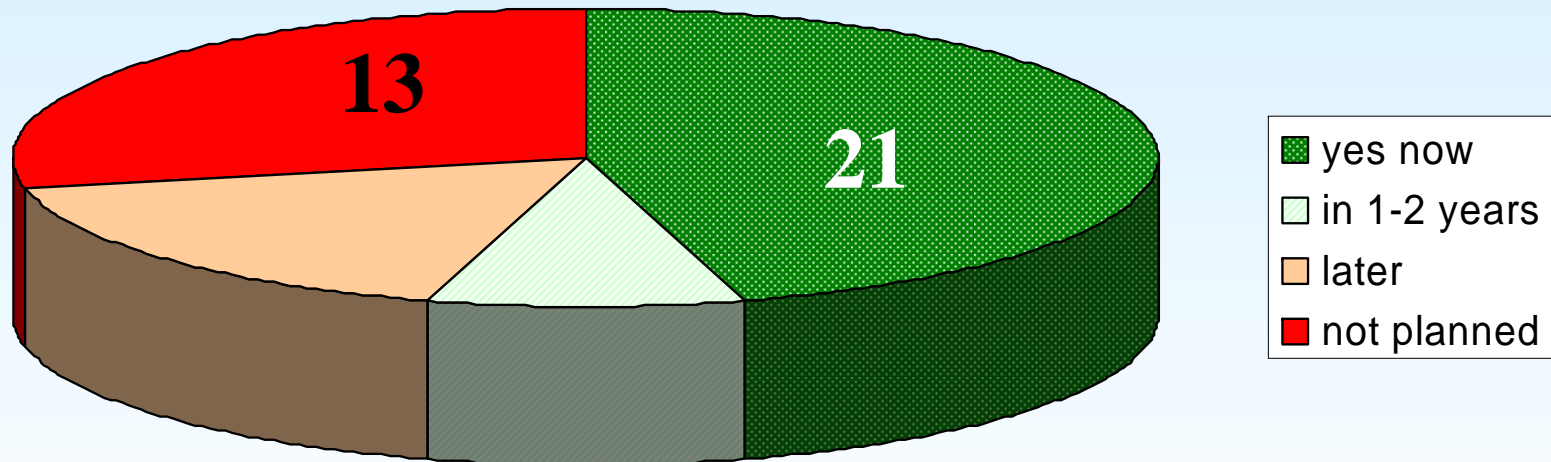
Nitrogen (n=29)

Company	Instrument	Number of Labs
CE Instruments	NC2500	1
	Flash EA-112	1
CFA Burkhard		1
Elementar	vario EL	6
	variomax CN	1
	variomax CNS	1
Gerhardt	Vapodest	4
Hekatech	CN	1
	EURO EA	1
LECO	CHN-2000	1
	CNS-2000	4
	CN-2000	5
	Truespec N	1
Thermo Finnigen	NCS Analyser Lash EA 1112	1



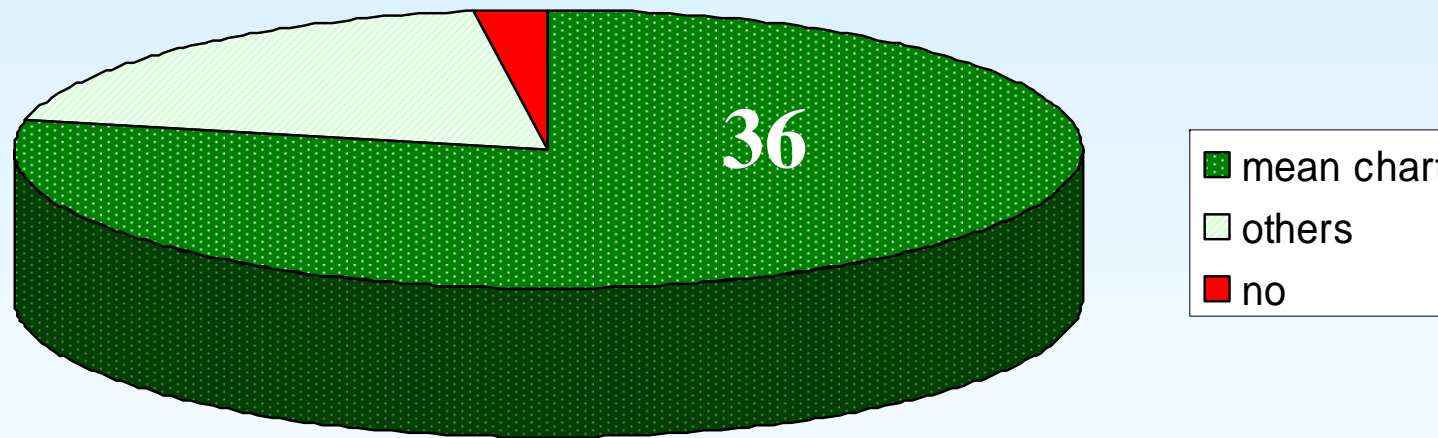
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Laboratory Accreditation (n=46)



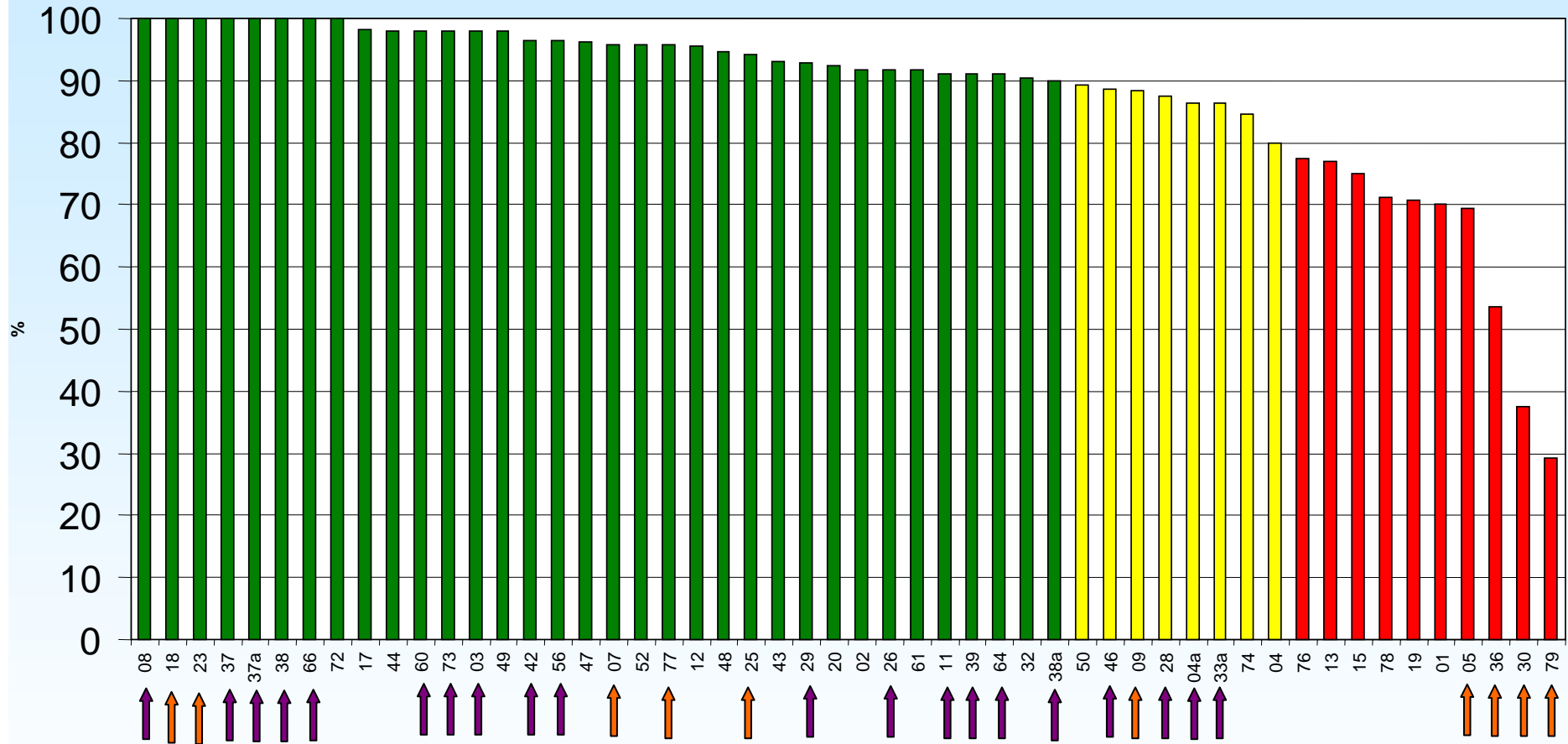
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Control charts (n=46)



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10th Ringtest Percentage of correct results



Laboratories with accreditation (for the foliage parameters)

Accreditation not planned



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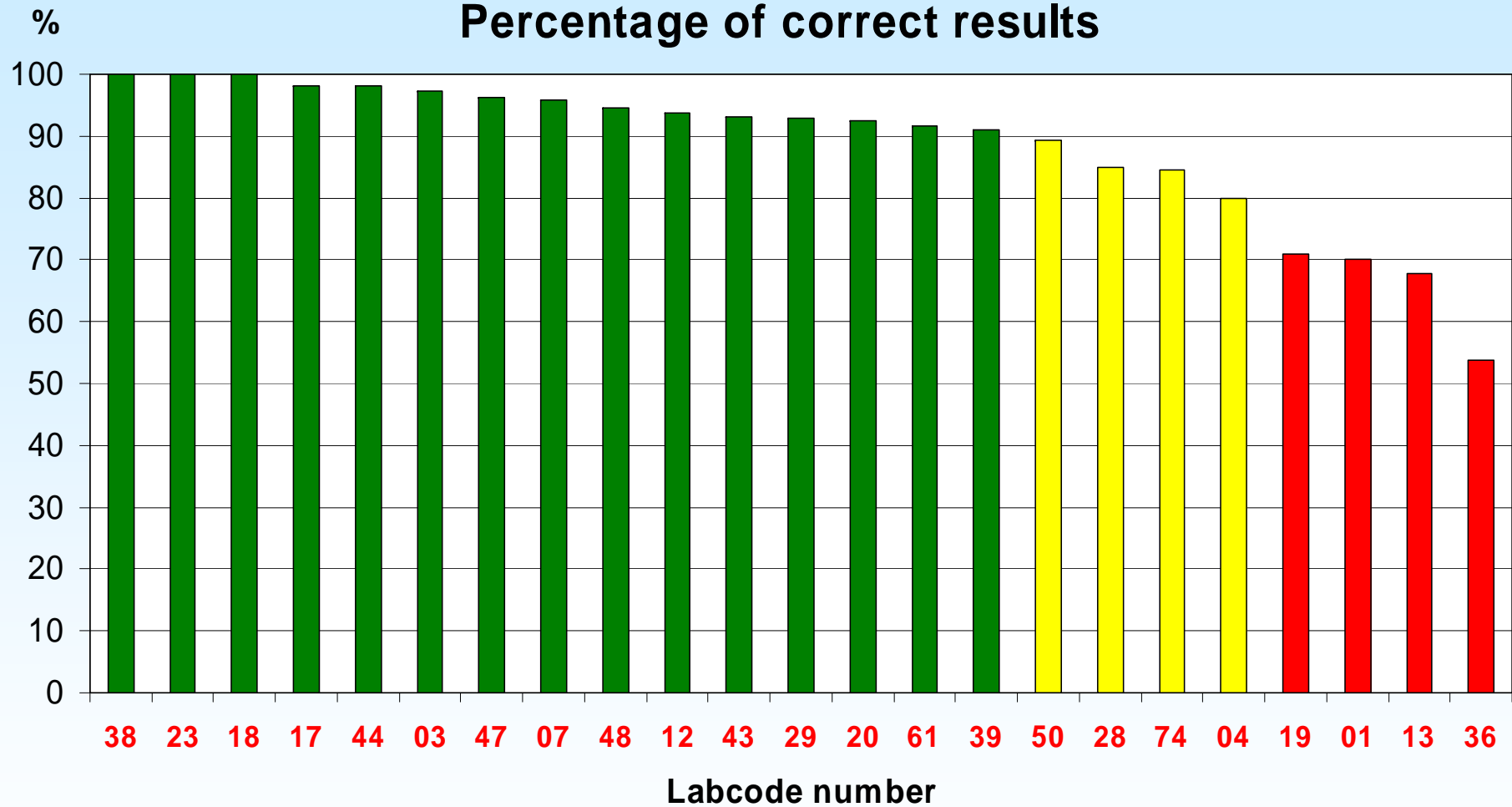
Which of these laboratories analyze Level II samples and submit results this year??

- Foliage level II survey is mandatory every two years (2007, 2009, 2011...)
- Litterfall should be sampled (and analyzed) continuously
- Different parameters for survey and ringtest
- Some labs are not participating in the Level II-surveys but in the ringtest programme
- **Participants will be asked when they save their results for each element**



10th Ringtest / "LEVEL II laboratories"

Percentage of correct results



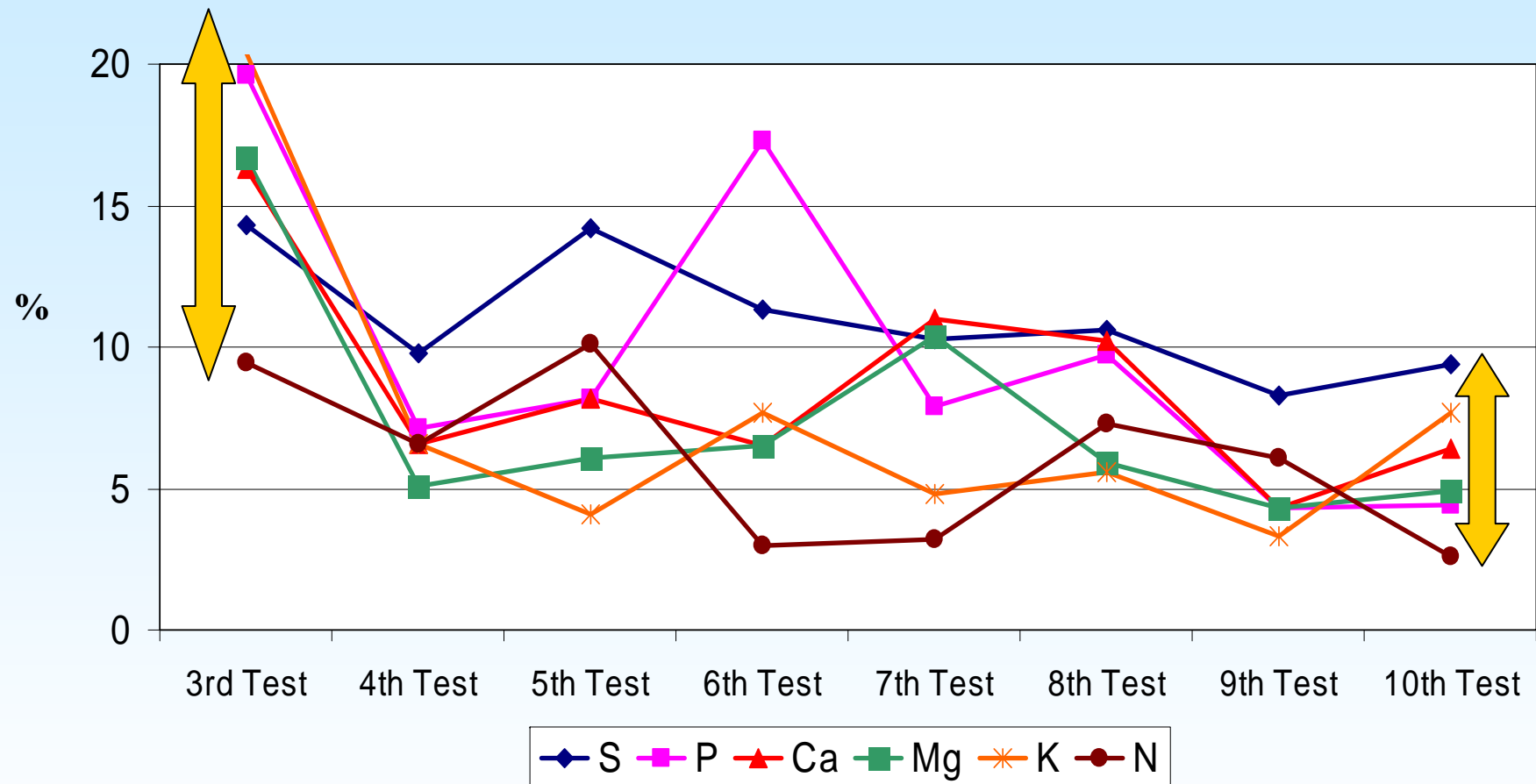
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Percentage of „non tolerable“ results

Element	+/-	8th	9th	10th
N	10	7.3	6.1	2.6
S	20/15	10.6	8.3	9.4/15.4
P	15/10	9.7	4.3	4.4/13.2
Ca	15/10	10.2	4.3	6.4/17.2
Mg	15/10	5.9	4.3	4.9/10.8
K	15/10	5.6	3.3	7.7/16.8



Progress in Quality - non tolerable results



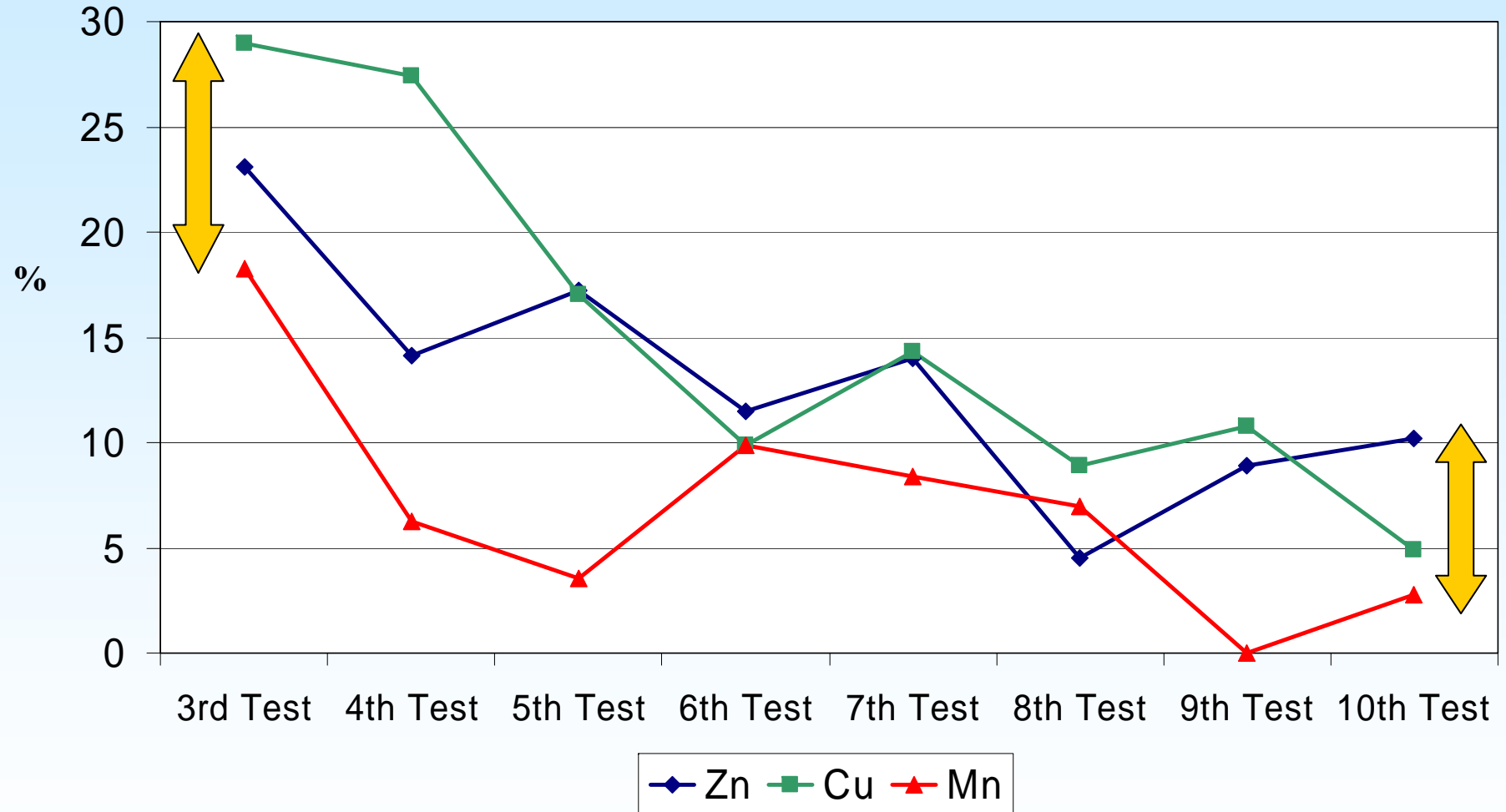
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Percentage of „non tolerable“ results

Element	+/-	8th	9th	10th
Zn	15	4.5	8.9	10.2
Mn	15	7.0	0.0	2.8
Fe	20	7.1	9.9	5.7
Cu	20	8.9	10.8	4.9
Pb	30	34.7	24.0	13.0
B	20	12.8	8.3	13.5
Cd	30	10.3	7.1	17.0
C	5	4.3	11.1	3.2



Progress in Quality - non tolerable results



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11th Needle/Leaf Interlaboratory Test (www.ffcc.at)

- 2008-03: Informing the laboratories
- 2008-07-04: **Registration** of the participants via internet
- 2009-01-04: **Deadline of data submission**

- **22 laboratories are registered** (9 labs participating in LIFE+) till 2008-04-01



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Future Tests – „correct units“

Magnesium [**mg/g**] - approximate range: 0.30 - 4.00 mg/g

	Replicate 1	Replicate 2	Replicate 3	Replicate 4
Sample 1 ()	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
Sample 2 ()	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
Sample 3 ()	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
Sample 4 ()	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
Pretreatment:	0 - No information <input type="button" value="v"/>			
Determination:	0 - No information <input type="button" value="v"/>			
Level II:	<input type="checkbox"/>			
	<input type="button" value="STORE RESULTS"/>	<input type="button" value="RESET FORM"/>	<input type="button" value="BACK"/>	



Last five Tests - some reactions about not satisfactory results

- It *was probably* a calibration problem.
- We did *our best this time* to fix the problem (and the results are still wrong.)
- *Hopefully* the problem was solved. (It was not solved.)
- We will contact you *soon*.... (but nobody will contact me.)
- We *know* the units are wrong - could *you* correct the results?
- We *know* we have problems with the instrument *but we sent* the results anyway.



Future Test – „Feedback“

- **Desires are no an useful feedback-facts are better!**
- Feedback had to includ: search and dedect the reason for the wrong results, set up milestones for improvement, reanalyzing the test samples & find the correct results
- Ask for feedback in case of not satisfactory results from **all** participants
- First feedback must be given within one month
- Short summary of the first feedback will be included in the final report



Proposal for tolerable Limits for lower Concentrations^{*)}

Element	Tolerable deviation from mean in %	concentration lower than
S	80-120	0.5mg/g
P	85-115	0.5mg/g
Mg	85-115	0.5mg/g
Zn	80-120	20µg/g
Mn	80-120	20µg/g
Fe	70-130	20µg/g
Pb	60-140	0.5µg/g

^{*)} Must be adopted from the EP Foliage and Litterfall



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Meeting of the Head of the Labs

- **Analytical Topics**

- QA/QC practice in a foliage laboratory - use of Reference Material & Control charts & Ringtests
- Reaction about not satisfactory ringtests results
- Flame AAS / Ca, Mg, K
- ICP-AES / Cd
- Calibration of Element-analyzer (C, N, S)
- List of candidates for the Assistance Programme

- **Other Topics**

- Feedback from the labs to FFCC about the ringtest implementation and improvement
- Who is a „Level II“ Laboratory and which lab is participating in FutMon?



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