Introducing tolerable limits for forest soil interlaboratory ringtests

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Tolerable limits

acceptable deviations (±) from the mean (nominal) value, expressed as a percentage of the mean

- Limits are defined for each variable and are method specific.
- Limits can be defined for a specific content or concentration range.
- Limits may be defined for each source of laboratory variance:
  - reproducibility variance (=between+within lab variance)
  - within laboratory variance
  - within run variance

Introduction

• For interlaboratory comparison of solid soil and forest floor samples, tolerable limits were calculated based on the Mandel's h (between laboratory variation) and Mandel's k (within-laboratory variation) statistics of the past FSCC soil ringtests

• An explanation of the evaluation methodology for the soil ringtests based on ISO 5725-2 is described in the FSCC ringtest reports (Cools et al. 2003, 2006, 2007).

The ringtest procedure allows the comparison of different sources of variance:

$$s_{Repr}^2 = s_{Lab}^2 + s_{Rep}^2$$

Where:

s_{Repr}^2 = estimation of the reproducibility variance
s_{Lab}^2 = estimation of the between-laboratory variance
s_{Rep}^2 = estimation of the repeatability (within-laboratory)

Methodology

Tolerable limits for the soil ringtests are inferred from the coefficient of variation for laboratory reproducibility CV_{Repr}

Coefficients of variation (CV)

Based on the general mean (Mgen) and the reproducibility variance (s_{Repr}), the coefficient of variation could be calculated.

$$CV_{Repr} = \frac{s\,\mathrm{Re}\,pr}{Mgen} \times 100 \qquad \text{as an estimate of } \frac{\sigma}{\mu} \times 100$$

Where:

sRepr = General standard deviation in the Mandels h/k plot
Mgen = General outlier free mean from the Mandels h/k plot

Analogue: CV_{Rep} based on sRep (Repeatability SD) / Mgen

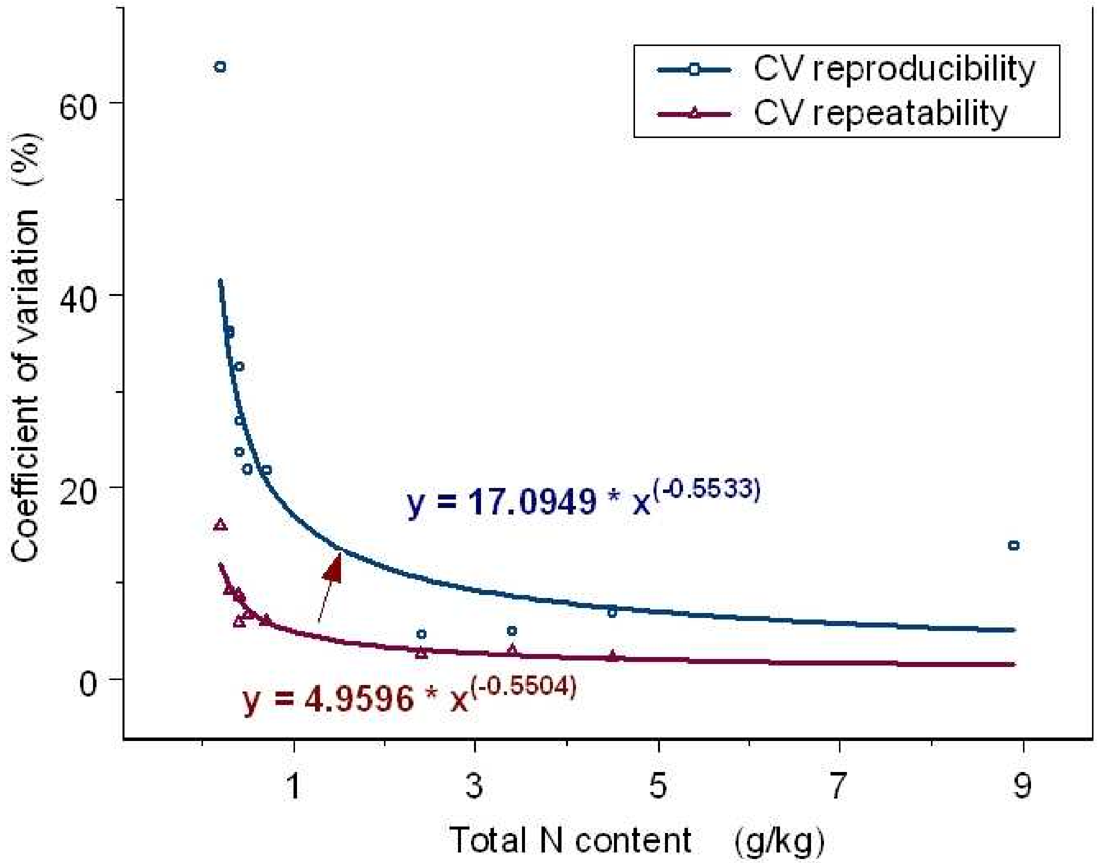
FSCC Ringtest results RT2-RT5

Ringtest	Code	Matrix	Origin	Soil type	Hor/Depth	Texture	Details
2nd (1995-1996)	2A	Mineral	Belgium	Podsol	B	Sand	Cover sand
2nd (1995-1996)	2B	Mineral	Belgium	Albeluvisol	Bt	Silt loam	Loess
3rd (2003)	3A	Mineral	Belgium		Ah	Sand	Chemically very poor forest soil
3rd (2003)	3B	Mineral	Belgium		Ah/E (0-10 cm)	Silty clay	Calcareous, eutrophic
3rd (2003)	3C	Organic	Belgium		F+H	Forest Floor	Same soil as 3A
4th (2005-2006)	4A	Mineral	Austria		20-40 cm	Silty clay	Relatively rich sample
4th (2005-2006)	4B	Mineral	Belgium		20-40 cm	Loam	
4th (2005-2006)	4C	Mineral	France	Calcic Luvisol		Sandy loam	Decarbonated topsoil
4th (2005-2006)	4D	Mineral	Germany			Sand	Chemically poor sandy soil
4th (2005-2006)	4E	Organic	Belgium		F layer	-	Forest Floor Beech
5th (2007)	5A	Mineral	Norway	Cambic Arenosol	Bs	Loamy sand	Same as Soil extract G from RT4
5th (2007)	5B	Mineral	Belgium		B (20-40 cm)	Loam	FSCC reference1
5th (2007)	5C	Mineral	Spain		C (7-29 cm)	Loam	Carbonate rich soil
5th (2007)	5D	Mineral	Germany	Colluvial brown earth	B (40-80 cm)	Loam	Sample also used in German RT
5th (2007)	5E	Organic	Sweden	Peat sample	-	-	Acid peat

15 real life test samples – 3 organic samples

Stepwise Methodology based on Ringtest results (RT2-RT5)

Step 1. Reproducibility and repeatability CVs of ring test samples are plotted against their content



n = 12 test samples

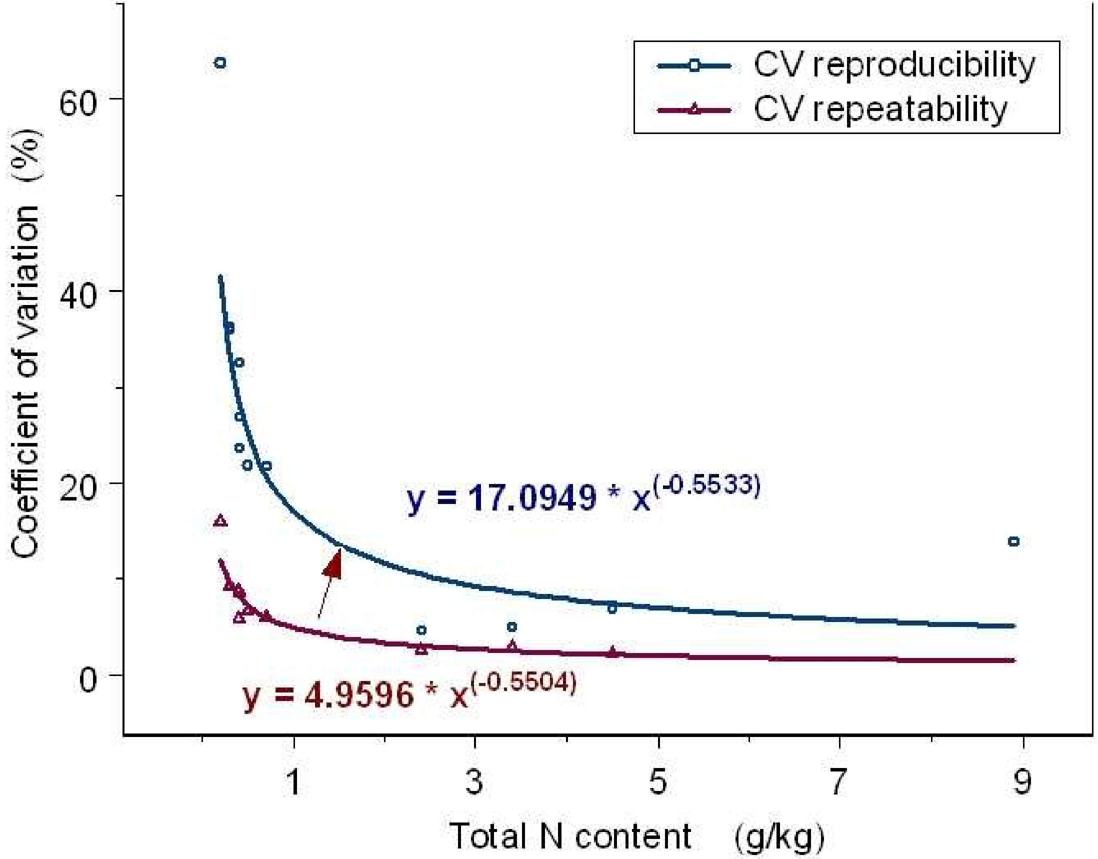
Range= 0.2 to 8.9 g kg^{-1}

It is well known that when contents are getting low, precision as expressed in the relative standard deviation (CV), gets worse.

Step 2. A power curve (y = a x^{-b}) was fitted to the ringtest results of each soil variable, for both the within lab repeatability and overall reproducibility

Methodology

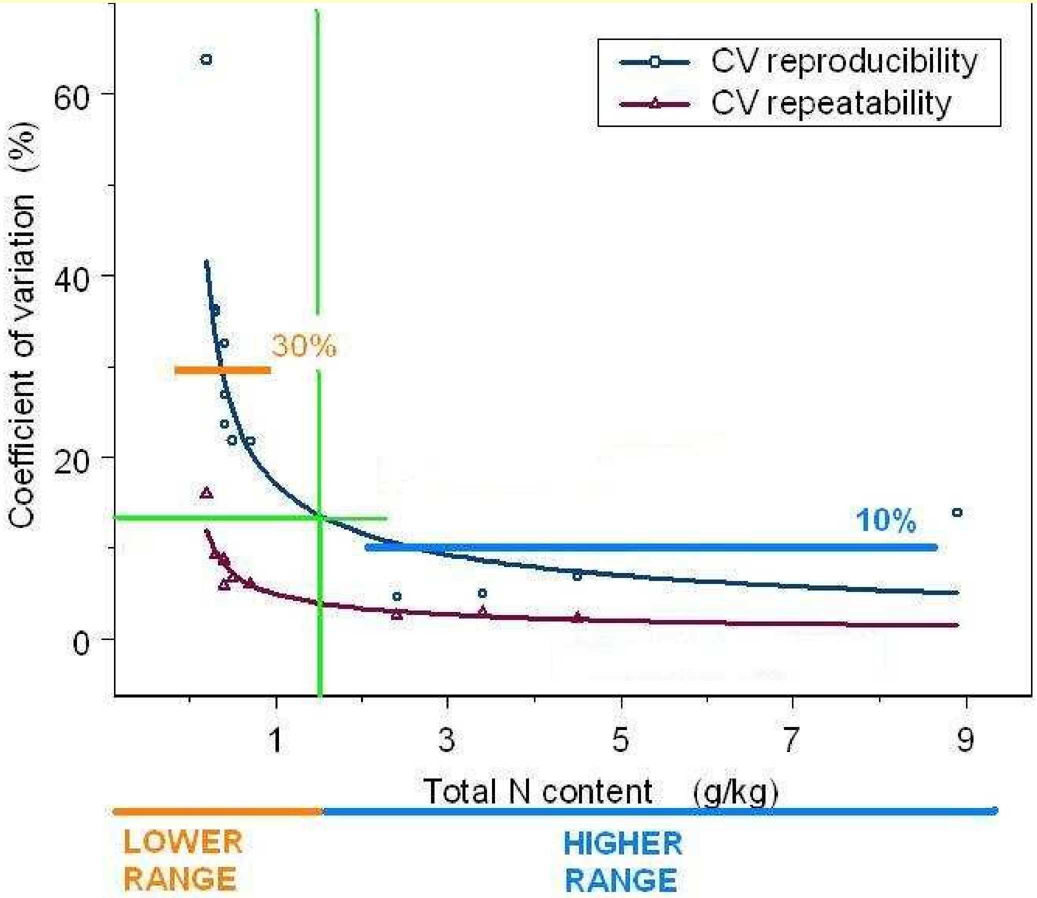
Step 3. Based on the reproducibility curve a bending point is set where a fast decrease changes into a slow decrease of precision.



This bending point splits the total determination range into a lower and a higher range

Methodology

Step 4. A lower and higher range is defined using the bending point. For both ranges, the average CV is calculated based on the experimental ringtest results, rounded to the nearest 5%.



Bend point @ 1.5 g kg^{-1} N

Range CVs repr.

Mean CV low= 31.9 ≈ 30%
Mean CV high= 7.7 ≈ 10 %

Range CVs repeatability

Mean CV low = 9 %
Mean CV high = 3 %

Methodology

Step 5. Average LOD and LOQ are predicted based on the repeatability curve fits using RSD values at 33% and 10% respectively

Estimated LOD and LOQ:

Solve $y = 4.9596\ x^{-0.5504}$ for

y = 33% => X = LOD = 0.04 g kg^{-1}

y = 10% => X = LOQ = 0.28 g kg-1

The average LOQ reported by the labs for total N determination was 0.1 g kg^{-1} nitrogen, which is between LOD and LOQ.

Note: LOQ definition is not standardized among labs ! (confidence level k differs 6, 10, …) For RSD 10% => k = 10 ! (IUPAC, 1997)

Methodology
defining the tolerable limits

Step 6. The outlier free mean for each test result is the mean value (nominal value) calculated using the statistical data evaluation procedure based on ISO 5725-2 *'Accuracy (trueness and precision) of measurement methods and results – part 2: Basic method for determination of repeatability and reproducibility of a standard measurement method'.*

Step 7. Tolerable limits expressed as percent of the mean, are inferred from the reproducibility CVs (z-score= 1; 1SD).

SD	% of labs within tolerable limits	
z-score	Theoretical	In practice (RT5 data)
1	68	70-80
1.15	75	80-90

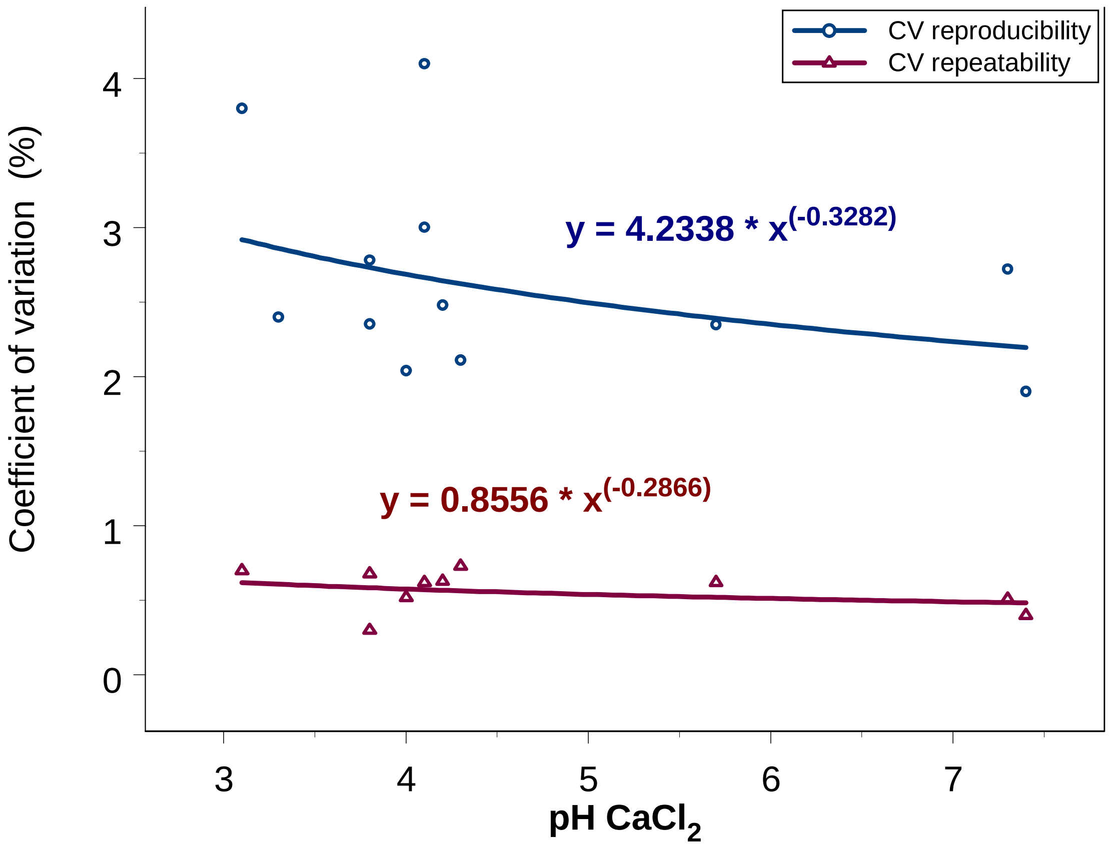
Therefore, initially we suggest to set the tolerable limits consequently to a z-score of 1 (= 1*SD), and round the ringtest limit values to the nearest 5% and the intra-laboratory limits to one percent.

Basic soil variables
suggested tolerable limits

Soil Property	Observation Range	Level	RingTest Tol. limit (% of mean)	Intra-laboratory Tolerable limit (% of mean)
Moisture content (%)	lower	**≤ 1.0**	**± 25**	**± 6**
	higher	**> 1.0**	**± 15**	**± 4**
pH_{H2O} -	whole	**2.0 – 8.0**	**± 5**	**± 1**
pH_{CaCl2} -	whole	**2.0 – 8.0**	**± 5**	**± 1**
OC g kg^{-1}	lower	**≤ 25**	**± 20**	**± 5**
	higher	**> 25**	**± 15**	**± 3**
N g kg^{-1}	lower	**≤ 1.5**	**± 30**	**± 9**
	higher	**> 1.5**	**± 10**	**± 3**
Carbonates g kg^{-1}	lower	**≤ 50**	**± 130**	**± 5**
	higher	**> 50**	**± 40**	**± 3**

pH_{CaCl2} results

pH in $CaCl_2$; n = 12; Range 3.1-7.4; CV < 4 % No bending point



The slope of a linear regression is not significantly different from zero with intercepts of 3.4 and 0.7 pH units for repr. and rep.

Hence no bending point is present and no lower or higher range is defined

Tolerance limit: ± 5%

For pH 5 = 0.25 pH u.

Soil labs report detectable difference of 0.02 pH u.

Is a CV_{repr} of 20% for Carbon too high ?

From ISO 10694 document – Total Carbon

An interlaboratory trial was organized in 1993 by the Wageningen Agricultural University, in the Netherlands, to verify the procedures specified in this International Standard.

Table A.1 — Types of soils used for the interlaboratory trial and their origin

Soil No.	Soil type	Origin
1	Muck soil	Poland
2	Andosol	Indonesia
3	Garden soil	Netherlands
4	Loess under forest	Switzerland
5	Sandy soil	Mali

Table A.2 — Results of the interlaboratory trial for the determination of organic carbon in soil

Parameter	Results				
	Soil No.				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	7	8	8	8	8
Number of outliers (laboratories)	—	—	—	—	—
Number of accepted results	—	—	—	—	—
Mean value (g/kg dry soil)	410,42	63,3	83,88	41,537	2,47
Standard deviation of the repeatability (s_r)	4,318	1,225	4,275	1,045	0,272
Relative standard deviation of the repeatability (%)	1,052	1,935	5,096	2,515	10,998
Repeatability limit ($r = 2,8 \times s_r$)	12,090	3,43	11,969	2,925	0,761
Standard deviation of the reproducibility (s_R)	127,413	11,957	19,376	5,523	1,555
Relative standard deviation of the reproducibility (%) →	11,087	18,888	23,098	13,297	62,92
Reproducibility limit ($R = 2,8 \times s_R$)	45,505	33,48	54,253	15,465	4,355

Soil texture suggested tolerable limits

Soil Property	Observation Range	Level	RingTest Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
Clay content %	lower	≤ 10.0	± **50**	± **8**
	higher	> 10.0	± **35**	± **4**
Silt content %	lower	≤ 20.0	± **45**	± **8**
	higher	> 20.0	± **30**	± **3**
Sand content %	lower	≤ 30.0	± **45**	± **6**
	higher	> 30.0	± **25**	± **2**

Exchangeable elements (1/2) suggested tolerable limits

Soil Property	Observation Range	Level	RingTest Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
Exch. Acidity $cmol_{(+)}\ kg^{-1}$	lower	≤ 1.00	± **90**	± **9**
	higher	> 1.00	± **35**	± **4**
Exch K $cmol_{(+)}\ kg^{-1}$	lower	≤ 0.10	± **45**	± **10**
	higher	> 0.10	± **30**	± **4**
Exch Ca $cmol_{(+)}\ kg^{-1}$	lower	≤ 1.50	± **65**	± **12**
	higher	> 1.50	± **20**	± **3**
Exch Mg $cmol_{(+)}\ kg^{-1}$	lower	≤ 0.25	± **50**	± **10**
	higher	> 0.25	± **20**	± **2**
Exch Na $cmol_{(+)}\ kg^{-1}$	whole	0.01-0.14	± **80**	± **14**

Exchangeable elements (2/2) suggested tolerable limits

Soil Property	Observation Range	Level	RingTest Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
ExchAl $cmol_{(+)}\ kg^{-1}$	lower	≤ 0.50	**± 105**	**± 12**
	higher	> 0.50	**± 30**	**± 4**
ExchFe $cmol_{(+)}\ kg^{-1}$	lower	≤ 0.02	**± 140**	**± 14**
	higher	> 0.02	**± 50**	**± 8**
ExchMn $cmol_{(+)}\ kg^{-1}$	lower	≤ 0.03	**± 45**	**± 7**
	higher	> 0.03	**± 25**	**± 6**
Free H+ $cmol_{(+)}\ kg^{-1}$	whole	0.02-1.20	**± 100**	**± 8**

Aqua regia extractable elements suggested tolerable limits (1/3)

Soil Property	Observation Range	Level	RingTest Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
Extr P mg kg^{-1}	lower	≤ 150	± **45**	± **3**
	higher	> 150	± **20**	± **3**
Extr K mg kg^{-1}	lower	≤ 500	± **60**	± **6**
	higher	> 500	± **40**	± **4**
Exct Ca mg kg^{-1}	lower	≤ 500	± **70**	± **7**
	higher	> 500	± **30**	± **3**
Exct Mg mg kg^{-1}	lower	≤ 500	± **60**	± **7**
	higher	> 500	± **15**	± **3**
Exctr S mg kg^{-1}	whole	35 - 1300	± **35**	± **4**
Extr Na mg kg^{-1}	lower	≤ 75.0	± **65**	± **8**
	higher	> 75.0	± **50**	± **6**

Aqua regia extractable elements suggested tolerable limits (2/3)

Soil Property	Observation Range	Level	RingTest Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
Extr Al mg kg^{-1}	lower	≤ 2500	± **50**	± **5**
	higher	> 2500	± **20**	± **3**
Extr Fe mg kg^{-1}	lower	≤ 2500	± **40**	± **4**
	higher	> 2500	± **15**	± **3**
Extr Mn mg kg^{-1}	lower	≤ 150	± **30**	± **4**
	higher	> 150	± **15**	± **4**
Extr Cu mg kg^{-1}	lower	≤ 5	± **40**	± **8**
	higher	> 5	± **15**	± **4**
Extr Pb mg kg^{-1}	whole	3 - 70	± **30**	± **4**

Aqua regia extractable elements suggested tolerable limits (3/3)

Soil Property	Observation Range	Level	RingTest Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
Extr Ni mg kg^{-1}	lower	≤ 10	**± 40**	**± 6**
	higher	> 10	**± 15**	**± 4**
Extr Cr mg kg^{-1}	lower	≤ 10	**± 40**	**± 7**
	higher	> 10	**± 25**	**± 4**
Extr Zn mg kg^{-1}	lower	≤ 20	**± 40**	**± 7**
	higher	> 20	**± 20**	**± 3**
Extr Cd mg kg^{-1}	lower	≤ 0.25	**± 100**	**± 5**
	higher	> 0.25	**± 55**	**± 6**
Exctr Hg mg kg^{-1}	whole	0 - 0.16	**± 75**	**± 6**

Reactive Al and Fe suggested tolerable limits

Soil Property	Observation Range	Level	RingTest Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
Reactive Al mg kg^{-1}	lower	≤ 750	± **30**	± **3**
	higher	> 750	± **15**	± **3**
Reactive Fe mg kg^{-1}	lower	≤ 1000	± **30**	± **4**
	higher	> 1000	± **15**	± **3**

Total elements suggested tolerable limits

Soil Property	Observation Range	Level	RingTest Tolerable limit (% of mean)	Intra-Laboratory TL (% of mean)
TotAl mg kg^{-1}	Lower range	≤ 20000	± **35**	± **4**
	Higher range	> 20000	± **5**	± **1**
TotCa mg kg^{-1}	Lower range	≤ 1500	± **20**	± **7**
	Higher range	> 1500	± **15**	± **2**
TotFe mg kg^{-1}	Lower range	≤ 7000	± **20**	± **5**
	Higher range	> 7000	± **5**	± **2**
TotK mg kg^{-1}	Lower range	≤ 7500	± **15**	± **3**
	Higher range	> 7500	± **5**	± **2**
TotMg mg kg^{-1}	Lower range	≤ 1000	± **60**	± **7**
	Higher range	> 1000	± **5**	± **2**
TotMn mg kg^{-1}	Lower range	≤ 200	± **25**	± **6**
	Higher range	> 200	± **5**	± **3**
TotNa mg kg^{-1}	Lower range	≤ 1500	± **20**	± **4**
	Higher range	> 1500	± **5**	± **2**

Tolerable limits preliminary conclusions

- Tolerable limits for all soil variables are suggested based on CV_{repr} and a z-score of 1

- Tolerable limits for higher ranges seem mostly acceptable 5-25 %, although some are >30 %

- Tolerable limits for the lower ranges are high to very high (up to 100% and more), revealing high between laboratory variability and measurement uncertainty

RIngtest Performance Amelioration Potential

Theoretically:

The RIngtest Performance Amelioration Potential (RIPAP) is defined as:

$$RIPAP = CV_{repr}/CV_{rep}$$

Indicator for the amelioration potential laboratories have to improve their analysis proficiency for a specific method

Ideally, RIPAP becomes 1 => all ringtest laboratories perform equally well for a specific method as one 'average performing laboratory'.

RIPAP ranges from 3-20

Methods with high RIPAP => **optimisation or replacement !**

The RIPAP ratio

Ranking of soil variables according to average RIPAP values based on FSCC ringtest performance (FSCC RT 2-5) set RIPAP >10

Variable	RIPAP
Carbonates	20.10
Extractable Cd	14.39
Extractable Hg	12.79
Free H+	12.48
Extractable P	10.67
Extractable K	10.51
Extractable Ca	10.41
Sand fraction	10.18
Extractable Al	9.01
Exchangeable Acidity	8.91
Clay fraction	8.58

Alternative method ?

Carbonate estimation based on total Ca & Mg content or exch Ca & Mg content

Labs are mostly measuring “noise” – is it relevant to measure these metals in forest soils systematically ?

Check method ! Improvement needed.

The RIPAP ratio

Ranking of soil variables according to average RIPAP based on FSCC ringtest performance (FSCC RT 2-5) – set RIPAP < 5.

Variable	RIPAP
Extractable Mn	4.93
pH_{H2O}	4.75
Extractable Cu	4.71
pH_{CaCl2}	4.50
Total Ca	4.40
Total Na	4.25
Moisture	4.11
Total Fe	4.09
Total Nitrogen	3.31
Total Mn	3.30
Total K	3.25

5th soil ringtest (Cools et al., 2007) provides data on 4 mineral soil samples (A-D) and one humus sample (E)

Code	Matrix	Origin	Soil type	Hor/Depth	Texture	Details
5A	Mineral	Norway	Cambic Arenosol	Bs	Loamy sand	Same as soil extract G from RT4
5B	Mineral	Belgium		B (20-40 cm)	Loam	FSCC reference1
5C	Mineral	Spain		C (7-29 cm)	Loam	Carbonate rich soil
5D	Mineral	Germany	Colluvial Brown Earth	B (40-80 cm)	Loam	Sample also used in German RT
5E	Organic	Sweden	Peat sample	-	-	Acid peat

Applying Tolerable limits on the 5th FSCC ringtest

TABLE results RT 5 report (outlier free mean results based on iterative procedure (ISO 5725-2) – see meeting documents

Parameter	Sample	A		B		C		D		E	
Parameter	Unit	N°	Mean	N°	Mean	N°	Mean	N°	Mean	N°	Mean
Moisture	%	35	0.9	39	0.7	39	1.7	39	1.2	38	7.5
Particle size clay	%	33	5.2	31	9.6	31	22.6	32	26.0		
Particle size sand	%	31	84.7	32	45.7	34	43.6	31	37.0		
Particle size silt	%	34	10.5	33	43.8	31	34.3	30	37.1		
pHCaCl2		45	4.2	41	3.8	43	7.3	44	4.0	37	2.8
pHH2O		43	4.6	44	4.2	45	7.9	45	4.6	45	4.0
CaCO3	g/kg					39	148.4				
OC	g/kg	38	8.0	41	6.7	40	27.9	32	3.8	38	497.4
Total N	g/kg	39	0.5	42	0.4	37	2.4	43	0.7	42	9.6
Exchangeable Acidity	cmol(+)/kg	32	1.43	29	3.23	8	0.12	32	1.88	26	6.93
Exchangeable Al	cmol(+)/kg	38	1.15	36	2.70	14	0.05	38	1.53	34	0.72
Exchangeable Ca	cmol(+)/kg	26	0.06	28	0.12	34	17.38	28	0.12	36	4.06
Exchangeable Fe	cmol(+)/kg	27	0.02	34	0.11	16	0.01	18	0.01	34	0.21
Exchangeable K	cmol(+)/kg	31	0.03	33	0.07	32	0.69	35	0.20	34	0.21
Exchangeable Mg	cmol(+)/kg	32	0.03	31	0.05	35	0.87	35	0.12	31	3.11
Exchangeable Mn	cmol(+)/kg	35	0.05	35	0.03	19	0.00	37	0.19	33	0.05
Exchangeable Na	cmol(+)/kg	17	0.02	24	0.04	22	0.04	23	0.02	26	0.27
Free H	cmol(+)/kg	13	0.05	23	0.23	5	0.06	17	0.10	25	5.61
Extractable Al	mg/kg	30	14568.0	31	8828.0	28	18281.3	27	25762.7	32	367.5
Extractable Ca	mg/kg	31	1699.1	34	350.7	33	76073.9	33	162.3	32	1081.3
Extractable Cd	mg/kg	21	0.106	15	0.046	21	0.161	18	0.116	22	0.193
Extractable Cr	mg/kg	38	24.6	37	20.7	37	24.1	34	37.2	24	1.4
Extractable Cu	mg/kg	37	11.6	37	4.5	34	13.2	35	12.3	30	1.8
Extractable Fe	mg/kg	32	21360.7	32	11709.2	33	20966.1	33	29367.1	32	397.8
Extractable Hg	mg/kg	12	0.019	14	0.030	11	0.026	12	0.024	10	0.038
Extractable K	mg/kg	32	1479.1	35	1581.2	34	4809.4	34	2414.0	32	114.1
Extractable Mg	mg/kg	30	3414.3	31	1346.0	36	3028.4	36	4200.7	35	500.1
Extractable Mn	mg/kg	38	437.9	37	109.3	37	360.3	34	1106.4	37	17.7
Extractable Na	mg/kg	29	92.4	28	51.8	31	141.4	28	113.3	30	94.3
Extractable Ni	mg/kg	34	18.7	31	4.9	35	17.3	34	58.7	23	1.1
Extractable P	mg/kg	34	688.6	33	101.3	34	428.9	36	279.9	35	182.3
Extractable Pb	mg/kg	38	8.0	38	8.0	34	13.3	36	11.5	34	12.5
Extractable S	mg/kg	26	91.3	24	77.0	25	418.7	25	131.8	25	1131.4
Extractable Zn	mg/kg	36	60.4	39	19.7	39	37.2	38	97.3	32	21.3
Reactive Al	mg/kg	24	2590.0	24	1372.1	25	771.3	24	1725.6	25	229.3
Reactive Fe	mg/kg	25	5637.4	25	2857.9	25	1763.3	24	3781.9	25	305.3
Total Al	mg/kg	10	47642.6	8	27010.0	10	42922.7	10	77065.5	9	666.9
Total Ca	mg/kg	9	8607.7	8	1430.6	10	76593.8	9	1322.2	6	1073.6
Total Fe	mg/kg	10	28129.9	10	13468.2	10	23571.2	10	35844.7	8	443.8
Total K	mg/kg	10	15145.5	10	13761.0	10	15974.9	10	26262.5	7	236.0
Total Mg	mg/kg	10	6818.8	8	1703.2	10	3785.4	9	6443.4	9	516.9
Total Mn	mg/kg	10	629.1	9	145.2	10	396.3	8	1148.0	9	20.0
Total Na	mg/kg	9	11705.3	10	5079.5	7	984.6	8	3303.0	9	147.9

Applying Tolerable limits on the 5th FSCC ringtest

- **Six groups of soil variables:**
  - **Basic soil variables (moisture, pH, carbonates, OC, N)**
  - **Texture variables**
  - **Exchangeable elements (incl. Exch Ac and Free H^+)**
  - **Extractable elements**
  - **Reactive Al & Fe**
  - **Total elements**

- For each group of soil variables the performance of the laboratories is evaluated, based on suggested tolerable limits.
- All labs that reported a value are evaluated, even those with extreme outliers.
- Using the outlier free mean content, the lower and higher limits of tolerance were calculated and the number and proportion of labs that reported average contents below, within and above the tolerable range were determined.

Example

RT5; sample A; Aqua regia extractable Zn content

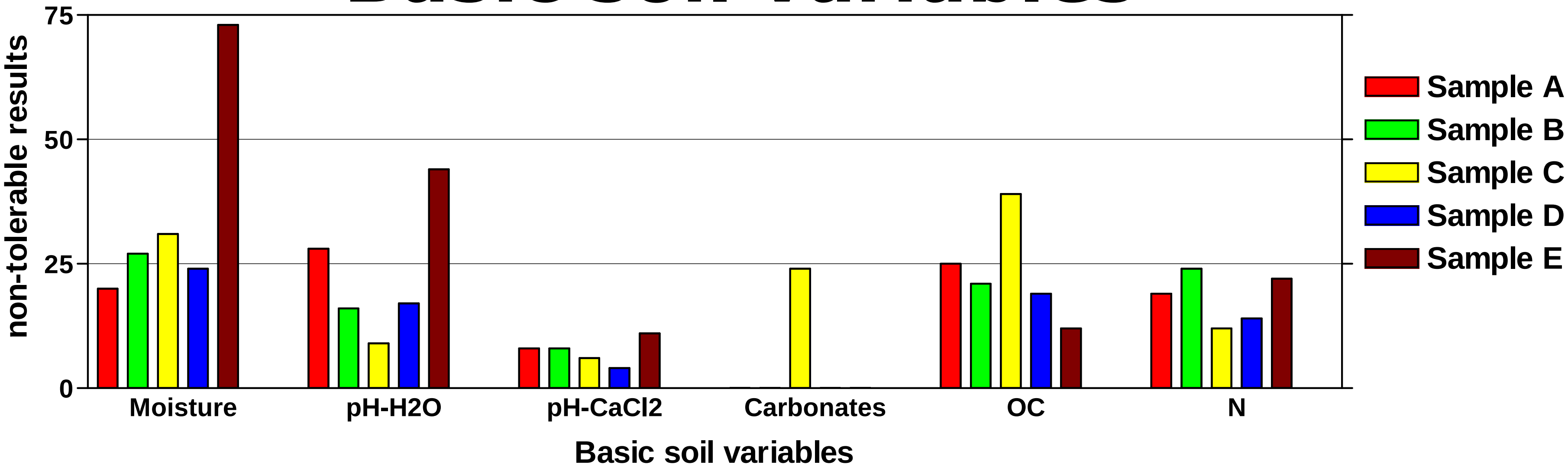
Submitted data from 41 labs:

9.13 44.33 46.67 46.87 50.06 50.27 51.87 55.15 55.70 55.83 57.50
57.70 59.03 59.17 59.33 59.47 60.07 60.40 60.47 60.70 60.80 60.93
60.93 61.00 61.70 61.87 62.20 62.60 62.60 63.26 63.33 63.90 63.91
66.00 68.63 68.67 71.77 72.36 77.00 82.67 330.97

Nominal value: **60.4 mg/kg**
Tolerable limits (for >20 mg/kg): **± 20 %**
Tolerable range 48.3-72.5 mg/kg

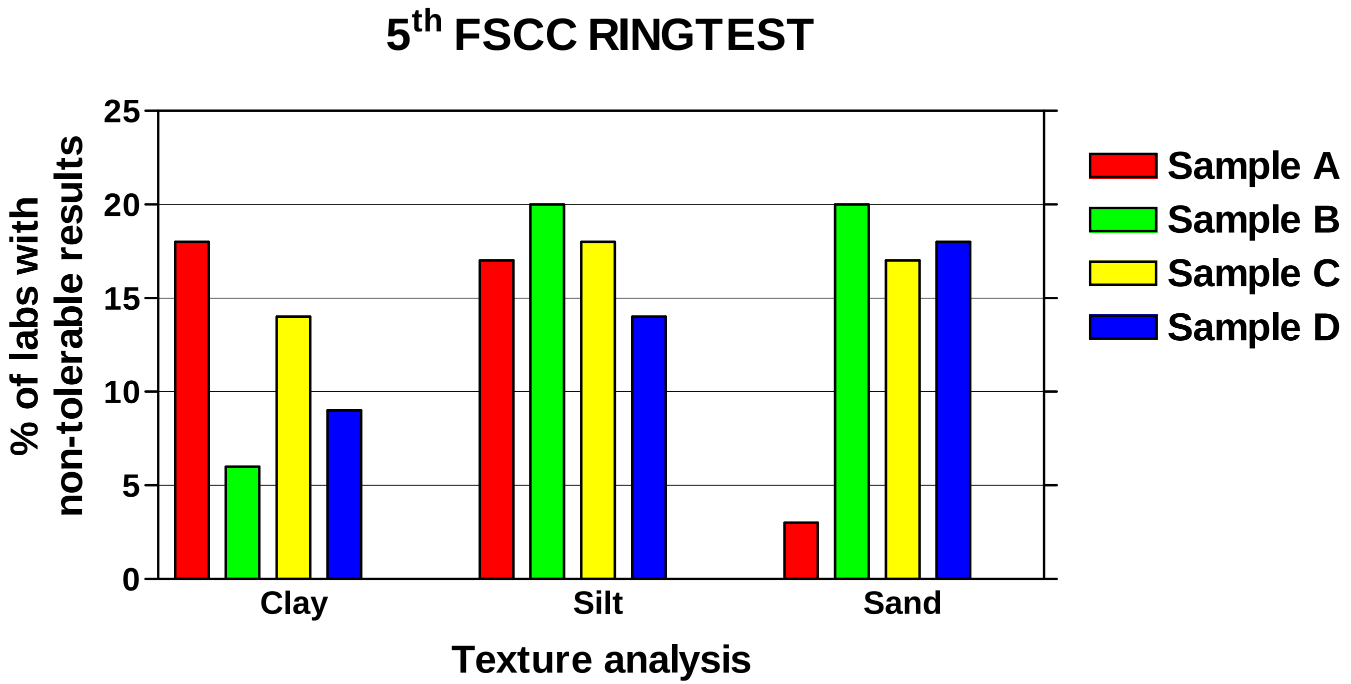
labs 4-34-3 or **10-83-7 %** of labs
% Labs with non-tolerable results: **10 + 7 = 17 %**

Basic soil variables



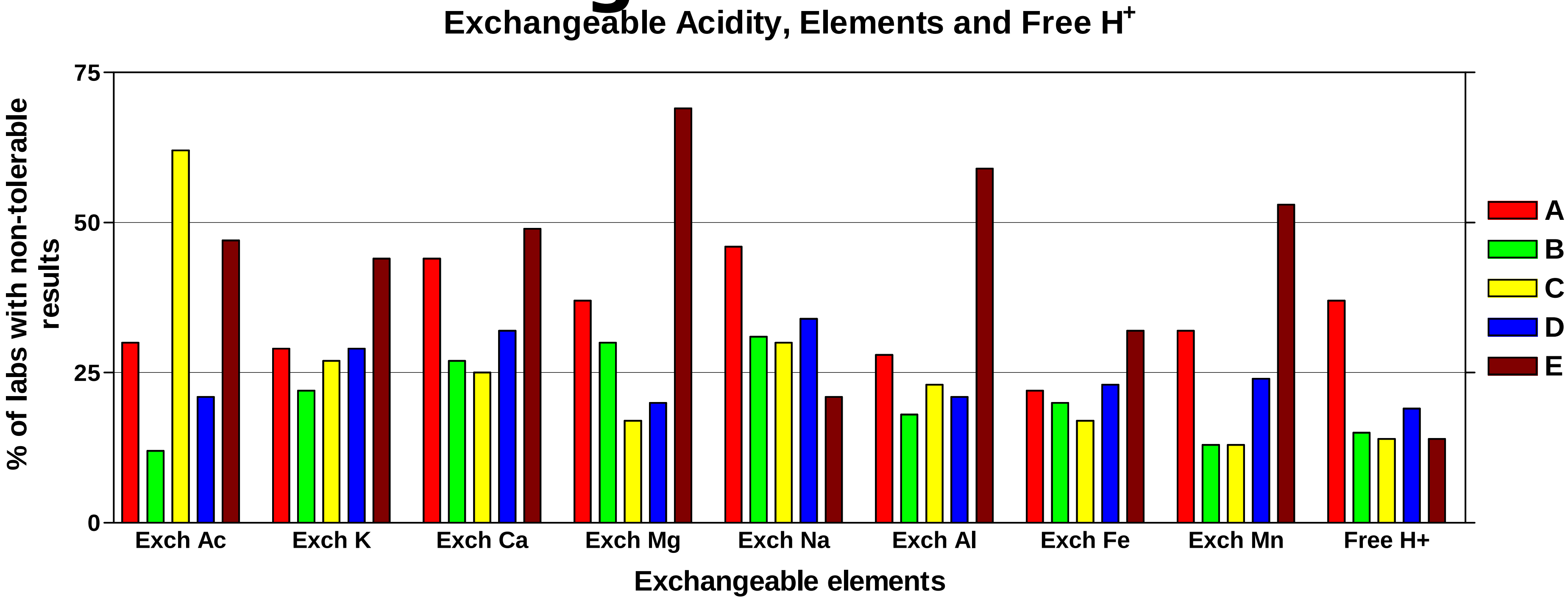
- For mineral soil samples (A-D) %non-tol results mostly **<25%**
- For peat sample (E) % non-tol results > 25 % for moisture content (not a ringtest parameter ! changing due to different (lab) environments !! Importance of this parameter for correction using mcf => impact on results ?)
- Results of FSCC reference material (B) are good indicator for average lab performance
- pH-$CaCl_2$ scores best (> 90% of labs within tolerable limits)

Texture variables



- Percentage of non-tol results below **20 %**
- Some variation among soil samples (fraction high => more labs within limits)

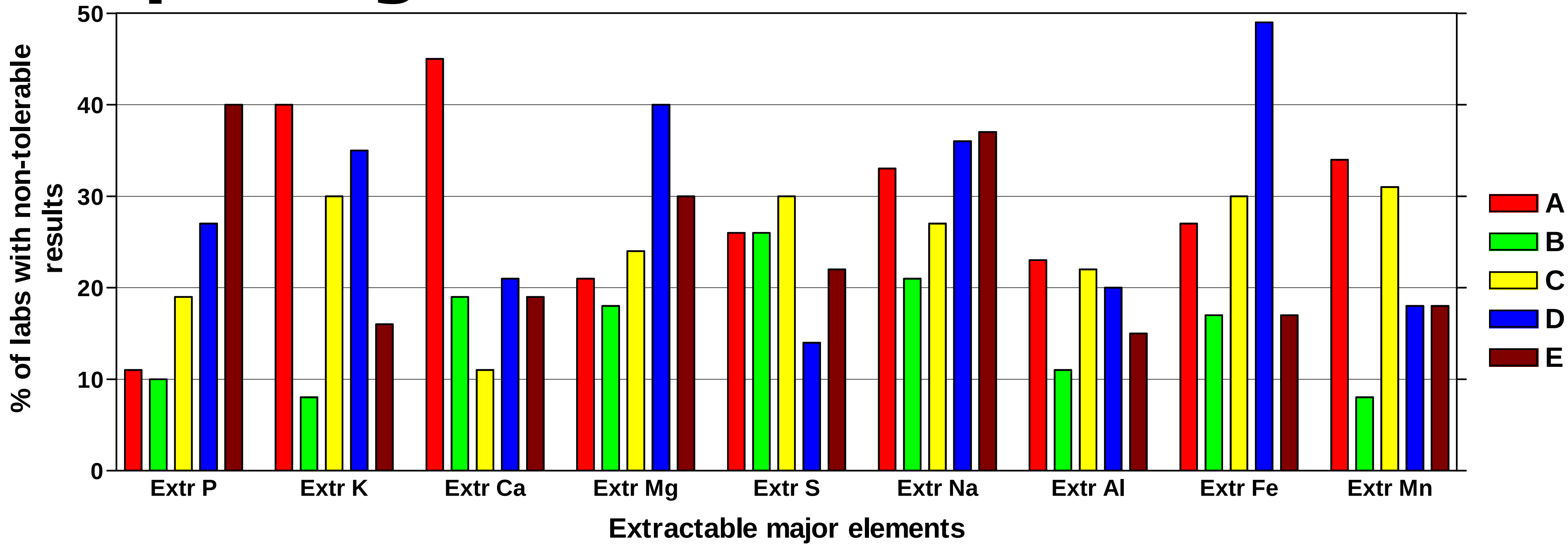
Exchangeable elements



- Despite the broad tolerable limits for lower levels of exchangeable acidity, only 38% of the labs report values within the tolerable range for sample C.
- More than 70 % of the labs report within the tolerable range for the other samples, except for the peat sample. The latter seems to be a difficult sample for determination of exch. elements.

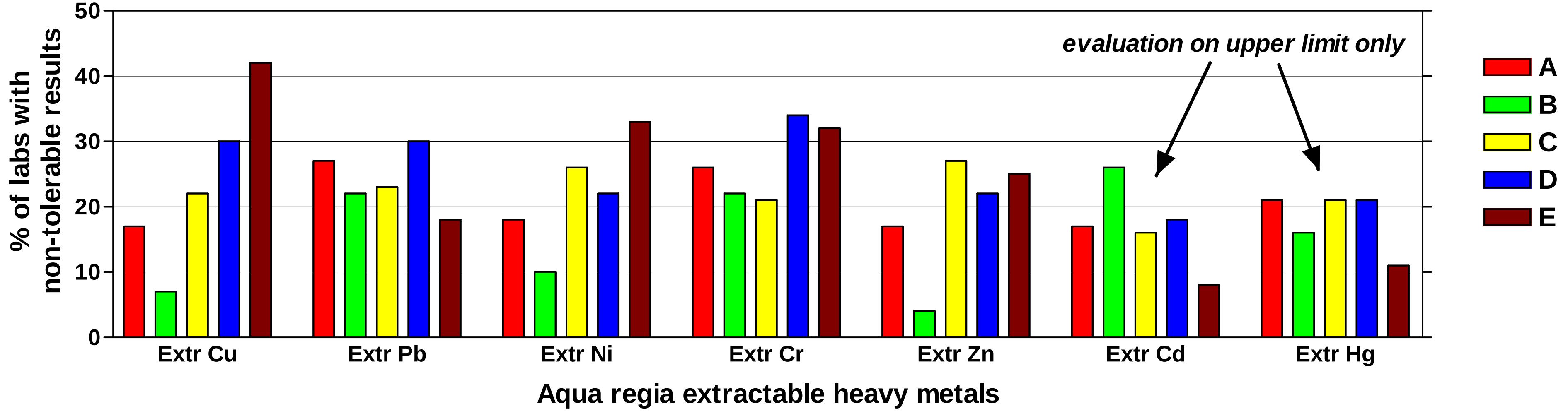
Applying Tolerable limits on the 5th FSCC ringtest

Aqua regia extractable elements (1/2)



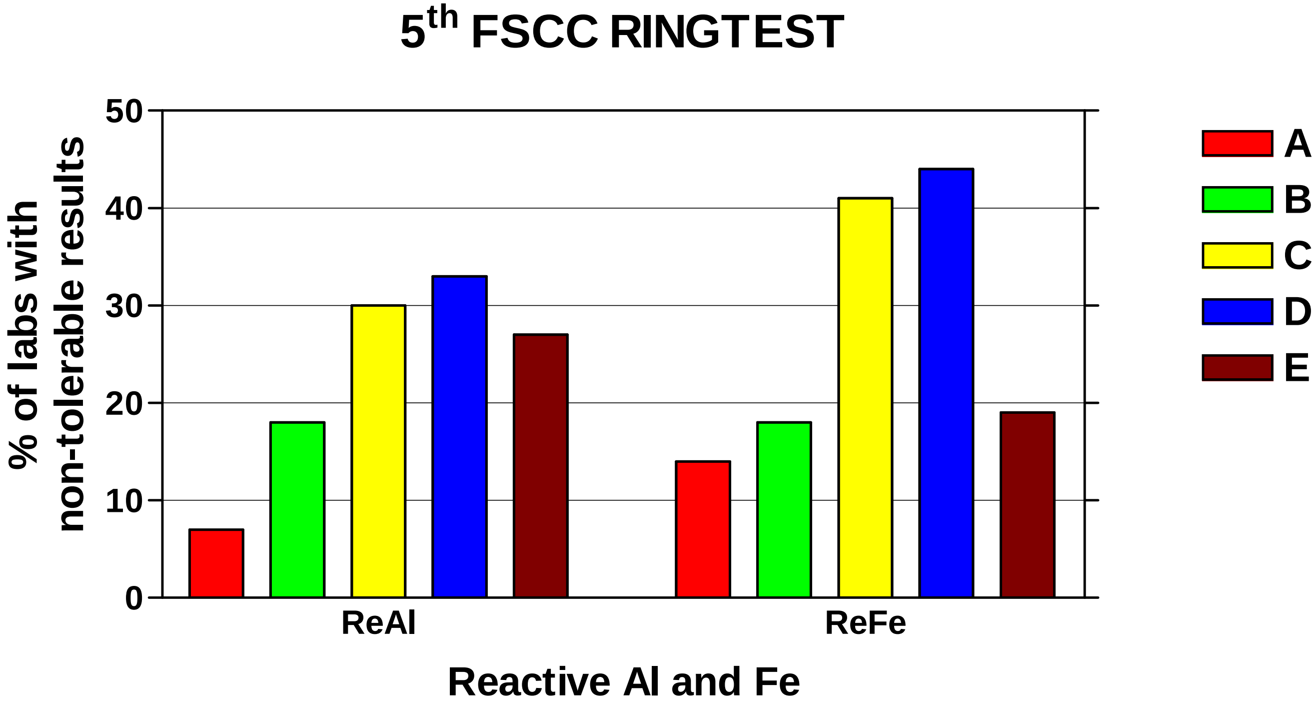
- For most elements the proportion of labs outside tolerable range is below 30%
- Some samples seem more difficult to analyse for specific elements: extr P & Na in Peat sample E, Extr Mg & Fe in D, …
- 80% of labs are capable of analysing these elements in the FSCC reference material within the tolerable limits

Aqua regia extractable elements (2/2)



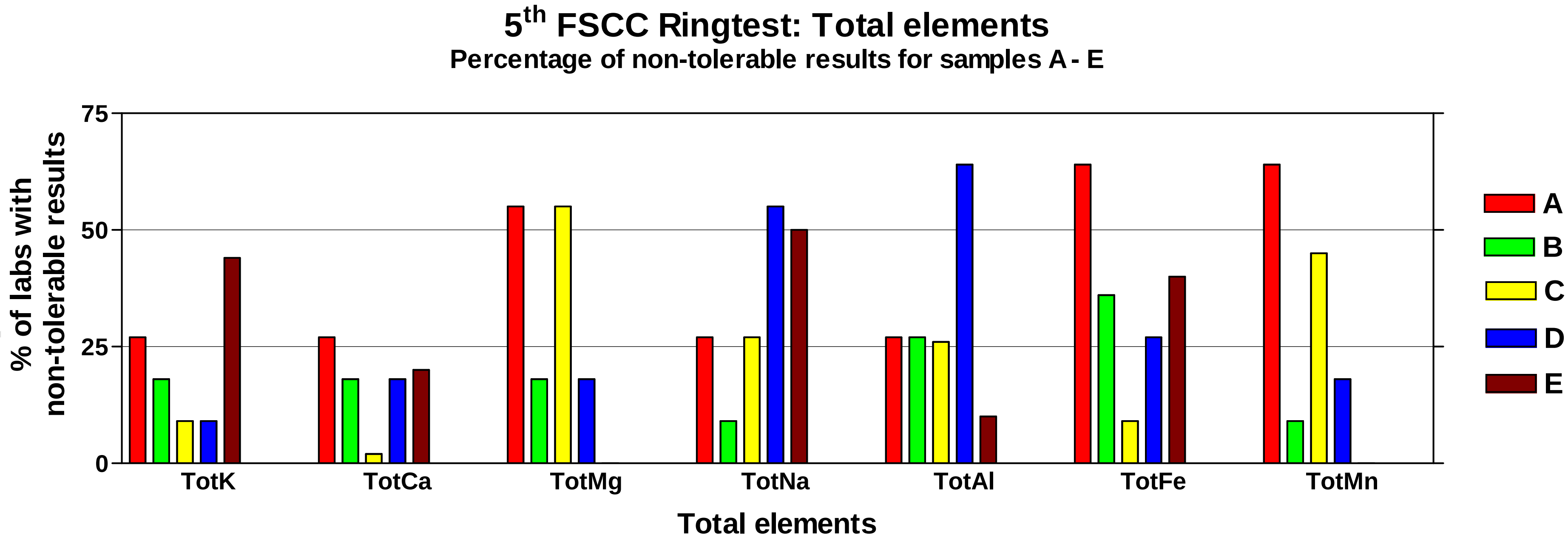
- For most elements the proportion of labs outside tolerable range is below 30%
- Lower tolerable limit for Cd and Hg may not be distinguished from “zero”, so only the upper tolerable limit is used for evaluationv

Reactive Al and Fe



- About one third of the labs failed in analysing reactive Fe and Al for samples C and D (however C low contents, D high)
- Most labs were within the acceptable limits for samples A & B (both high contents)

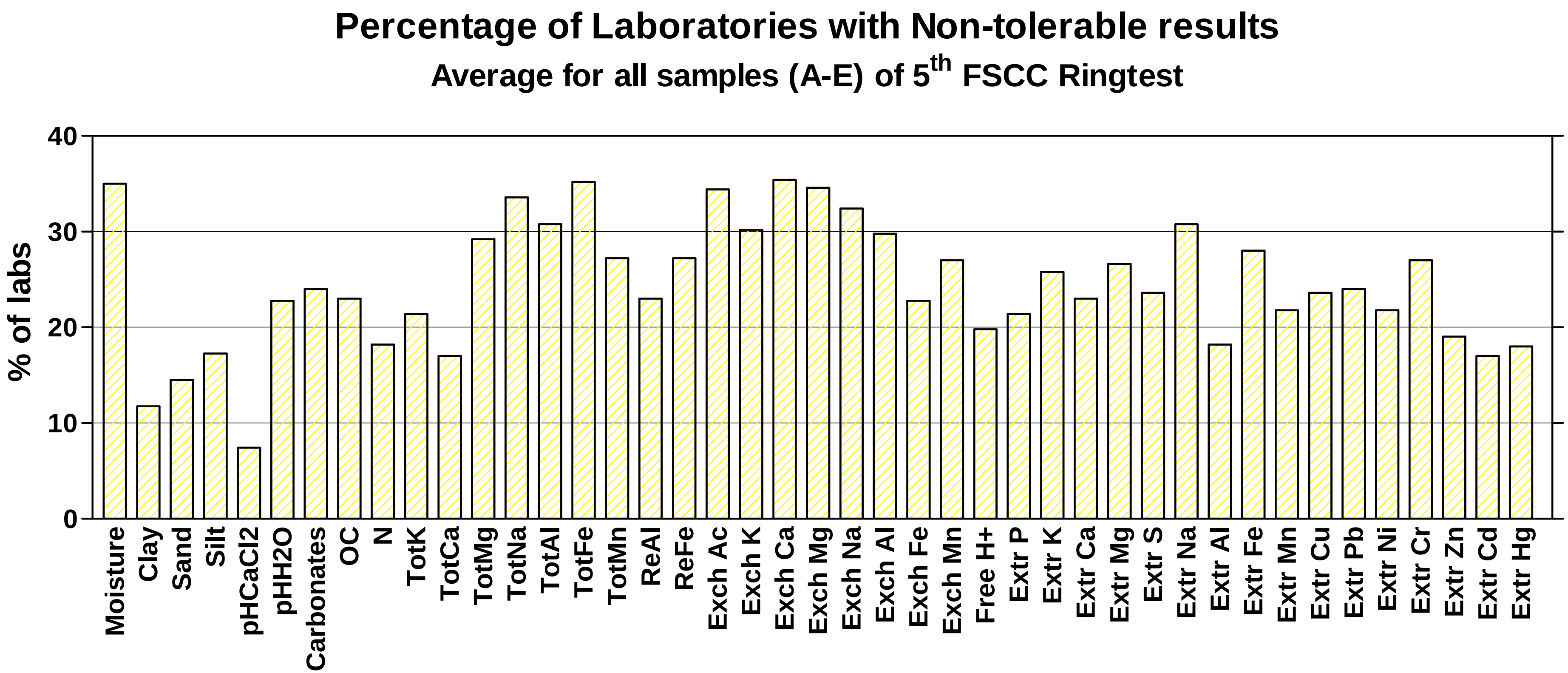
Total elements



- High variation of lab performance among samples !
- Different pattern per element
- For some elements and samples > 50 % of labs fail

Applying Tolerable limits on the 5th FSCC ringtest

General performance



For all parameters: % of labs with non-tol results range from 7.4 to 35.4 % with an overall average of 25 % of the labs

Some conclusions & questions:

- On average for all variables: three out of four labs fell within the tolerable range for FSCC RT5
  or 25% failed. Can we live with that ?

- Some samples are clearly more difficult to analyse than others (f.i. peat sample)

- Differences in reported soil moisture (air to oven dry mass) are large, and consequently an important factor. How is this affecting all reported results ? Do laboratories consequently apply a mcf to express their results on 105°C ? If they analyse from air-dry samples, do they always have the same moisture content prior to analysis and is their used mcf representative ? (error impact up to 3.7 % ?)

- Based on the RIPAP ratio some methods should be revised or discussed if their systematically application is still relevant !