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1st FSCC Soil Physical Ring Test

Further development and implementation of an EU-Level Forest Monitoring System (FutMon), Life+ Regulation of the European Commission, in cooperation with the International Cooperative Programme on Assessment and Monitoring of Air Pollution Effects on Forests (ICP Forests)

Nathalie Cools, Bruno De Vos

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LIFE+07 ENV/D/000218/Action C1-Soil-3(FL): Quality, expertise and evaluations within soil surveys

Vlaamse overheid

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FURTHER DEVELOPMENT AND IMPLEMENTATION OF AN EU-LEVEL FOREST MONITORING SYSTEM (FUTMON)

AND

UNITED NATIONS ECONOMIC COMMISSION FOR EUROPE

CONVENTION ON LONG-RANGE TRANSBOUNDARY AIR POLLUTION

INTERNATIONAL COOPERATIVE PROGRAMME ON ASSESSMENT AND MONITORING OF AIR POLLUTION EFFECTS ON FORESTS (ICP FORESTS)

1st FSCC Soil Physical Ring Test 2009

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Summary

Twenty-three laboratories participated in this first FSCC Soil Physical Ring Test. Based on the ISO methodology for the determination of repeatability and reproducibility of a laboratory measurement method four laboratories (A66, P02, P11 and P12) were excluded for at least one of the nine evaluated parameters. Eight laboratories (F03a, F12, F15, F27, P06, P14, S04 and S22) passed easily the ring test without any outlier, straggler or tail value for any of their reported parameters.

One laboratory (P12) was excluded based on poor between laboratory reproducibility for five of its reported parameters. Laboratories P11, P02 and A66 showed poor repeatability for some parameters because of one deviating subsample.

In all, the results of this ring test are very good. Six of the nine reported parameters show a coefficient of variation below 10%. Especially at the lower matric potentials, we do not expect a considerable quality improvement when all the analyses would be performed by one central laboratory. The problems are mainly situated in the measurement of the volumetric water content at pressures of -250 and -1500 kPa. Since the latter is a critical value for estimating the available water capacity of soils, improvement in the interlaboratory comparability at the higher matric potentials remains an important challenge.

Concerning the methods, it still needs to be clarified whether the ceramic pressure plate extractors can be used for pressure heads -1, -5, -10 and -33 kPa as this seems to be a common practice while it is not accepted by the present FutMon field protocol and by ISO11274 (ISO, 1998). It is beyond all discussion, that the volumetric water content needs to be reported at fixed pressure heads in order to come to a harmonised forest soil water retention curve database at the European level.

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1 Introduction

The soil water retention characteristic (SWRC) is the relationship between volumetric soil water content and the matric pressure. It depends on soil texture, organic matter content, bulk density and varies vertically and horizontally in the soil profile. The determination of the SWRC is essential for the comparison of different water balance models in the Life^{+} FutMon action C1-Met-29(BY) 'Quality, expertise and evaluations within meteorological surveys'. On more than 100 IM2 plots, these measurements will be carried out in the D3 action on 'Water budgets'. The SWRC will be measured for specific soil layers for at least 3 profiles on each D3 plot. Based on the water content measured in the field and the SWRC in the lab, the matric potential for each layer is inferred.

Within action C1-Soil-3(FL) the Forest Soil Co-ordinating Centre (FSCC) developed the protocol SA14 (2009) on soil water retention measurements in the laboratory. To assure the quality within this new survey, all 22 associated beneficiaries participating in action D3 had to take part in an intercalibration exercise of SWR data. This report discusses the results of this 1st FSCC Soil Physical Ring Test.

This ring test was mandatory for all laboratories taking part in action D3 and was optional for other laboratories participating in the ICP Forests monitoring programme. All registered labs had to follow the time table:

- Registration by the 27th of March 2009
- Registered laboratories sent their own empty steel rings (plus caps) to FSCC till the end of March 2009
- Labs received their own steel rings with compacted soil for SWRC analysis by the 15th of April 2009
- The method for analysis was described in the FutMon protocol SA14 (based on ISO 11274) Version 1.2.
- Labs submitted their retention data according to a fixed reporting format till the 15th of August 2009 to FSCC by email (fscc@inbo.be)
- The report with the statistical analysis was available at the FSCC website by end of September 2009. The same ISO:5725-2 methodology as for chemical soil ring tests was used for evaluation.
- The results of the 1st FSCC Soil Physical Ring Test were discussed at the Meeting of the heads of the labs, Warsaw, 12-13 October 2009.

The primary aim of the ring test is to gain insight in the statistical differences in soil moisture retention measurements due to the fact that analyses are conducted in different laboratories across Europe. Secondly, the variation between five replicates of the same sample analysed within one laboratory is assessed. So this ring test assessed two sources of variance: the between and within laboratory variance.

2 Materials and methods

2.1 Selection and registration of the laboratories

According to the FutMon proposal and in line with the outcome of the kick-off meeting of the Life$^+$ FutMon project on 12-16 January 2009 in Hamburg, all laboratories which analyse samples (either on deposition, soil, soil solution, soil physics, foliage, litterfall or ground vegetation) had to take part in a number of ring tests during the two project years, amongst others the 1st FSCC Soil Physical Ring Test 2009. All associated beneficiaries provided contact details of the participating laboratories to the chair of the Working Group of QAQC in the laboratories. The laboratories received their new lab code which was harmonised for the different ring tests in the project. Registration was possible by the end of March 2009.

Together with the registration, the laboratories were asked to send FSCC five identical steel core cylinders with their respective lids for sampling the "undisturbed' ring test sample. It was decided to use the cylinders that the laboratories are familiar with and for which each laboratory is equipped. These cores should also be the cores that the laboratories will analyse during the FutMon survey.

Countries participating within the ICP Forests programme without being associated beneficiary of the FutMon project were invited to take part in the ring test on a voluntary basis.

2.2 Basic physico-chemical characteristics of the test sample

The soil material which has been used to fill the soil cores is a subsample of the FSCC soil reference material. The soil properties and the element concentrations are given in Table 1. The material is taken from an acid loamy forest soil at 20 – 40 cm depth. The dry soil bulk density was 1474 ± 97.5 kg.m^{-3}.

Table 1: The chemical characteristics of the test sample

Particle size distribution			Exchangeable cations			Aqua regia extractable elements					
Clay	9.5	%	Al	2.85	cmol(+)/kg	Al	9017	ppm	Mg	1348	ppm
Silt	48.6	%	Fe	0.10	cmol(+)/kg	Ca	354	ppm	Mn	113	ppm
Sand	41.2	%	Mn	0.032	cmol(+)/kg	Cd	0.06	ppm	Na	48.0	ppm
			Ca	0.11	cmol(+)/kg	Cr	22.0	ppm	Ni	5.14	ppm
Soil reaction			K	0.065	cmol(+)/kg	Cu	4.61	ppm	P	105	ppm
pH(H_2O)	4.24		Mg	0.046	cmol(+)/kg	Fe	11610	ppm	Pb	8.57	ppm
pH($CaCl_2$)	3.84		Na	0.032	cmol(+)/kg	Hg	0.029	ppm	S	76	ppm
			Free H+	0.17	cmol(+)/kg	K	1641	ppm	Zn	20.2	ppm
Total Organic carbon	6.38	g/kg	Acidity	3.21	cmol(+)/kg						
						Reactive elements					
Total Nitrogen	0.44	g/kg				Al	1317	ppm			
						Fe	2764	ppm			

2.3 Sample preparation

A semi-artificial soil sample was tested in this interlaboratory ring test. After profound homogenisation by quartering, the loose and moist sample was sieved on a sieve with mesh size of 1 cm to reduce the stone content. Then a layer of 40 cm soil was brought into a steel container (48 cm wide and 48 cm length). Subsequently the sampling cores were carefully brought into the soil and again covered by a layer of 20 cm soil. Then the soil was artificially compressed using a hydraulic compression system using a maximal pressure of 120 bar driven by the PTO of a tractor.

Photo 1: Hydraulic compression system driven by the PTO of a tractor

Photo 2: Steel container filled with sample material in which the cores are imbedded

Then the samples were carefully dug out by hand. The soil was cut by an iron saw to the upper and lower edge of the rings. The rings were sealed with the lids provided by the laboratories and wrapped into plastic foil. Then the rings were carefully packed and sent by international courier to the registered laboratories.

Though, when the samples reached the laboratories, many of the samples showed cracks due to physical disturbance and/or desiccation during (air) transport. See some examples below. Based on this reason, laboratory F11 decided to withdraw from the ring test.

Possibly the damage would have been less if the samples would have been more moist and closer to field capacity. This would of course increase the transportation costs since the samples would have been heavier. However by simply saturating the soil samples, the soil should fit nicely again in the steel cores.

Photo 3: One sampling ring of lab P11 where the soil shrunk during transport

Photo 4: Three sampling rings of lab S04 showing cracks by disturbance

2.4 Homogeneity tests

A few homogeneity tests were carried out prior to the sampling for the ring test. The variation in the dry soil bulk density was compared within one container for different core sizes and between three different containers. In each container five rings of 100cc and five rings of 300cc were placed. No significant differences in bulk density between the containers, neither between the ring types was found. The mean bulk density was 1408 $kg.m^{-3}$ with a coefficient of variation of at most 3.92%.

Photo 5: Homogeneity tests in the containers

2.5 Distribution of the samples and submission of results

The five replicates of the test sample were sent to the registered laboratories by the 6th of April 2009. The laboratory results had to be reported to FSCC by the 15th of August 2009 following the format and precision requirements of the FutMon protocol SA14. See Table 2 below. However, several laboratories faced particular problems and asked for postponement. This report includes all results that FSCC received by the 11th of September 2009. This is four weeks after the initially agreed deadline.

Table 2: The list of matric pressure heads at which the volumetric water content is to be measured with specifications of units and numeric precision (after protocol SA14)

Matric pressure (kPa) ψ	Volumetric water content (VWC) = θ	unit	Numerical Precision
0	0.xxxx	$m^3\ m^{-3}$	0.0001
-1	0.xxxx	$m^3\ m^{-3}$	0.0001
-5	0.xxxx	$m^3\ m^{-3}$	0.0001
-10	0.xxxx	$m^3\ m^{-3}$	0.0001
-33	0.xxxx	$m^3\ m^{-3}$	0.0001
-100	0.xxxx	$m^3\ m^{-3}$	0.0001
-250	0.xxxx	$m^3\ m^{-3}$	0.0001
-1500	0.xxxx	$m^3\ m^{-3}$	0.0001
Matric pressure (kPa) ψ	Dry bulk density (BD)	unit	Numerical Precision
-10^6	xxxx	$kg\ m^{-3}$	0

2.6 Laboratory Analytical Methods

Laboratories were asked to use the methods as described in the FutMon protocol SA14, Version 1.2 including suggestions and corrections after the FUTMON –D3 Soil moisture workshop in Freising on 25-26 March 2009. See Table 3. This protocol follows the ISO 11274 (1998).

Table 3: Recommended instructions for the volumetric water determinations at predefined matric potentials

Matric potential ψ		Mandatory /Optional	Recommended instrument	Estimator
pF	kPa			
0.0	**0**	**M**	**Sand suction table**	**≈θsat= Water holding Capacity (WHC)**
1.0	**-1**	**M**	**Sand suction table**	
1.7	**-5**	**M**	**Sand suction table**	
2.0	*-10*	*O*	*Sand suction table*	*Field Capacity sand*
2.5	**-33**	**M**	**Kaolin suction table**	**Field Capacity siltloam**
3.0	*-100*	*O*	Kaolin suction table	*Field Capacity clay*
3.4	*-250*	*O*	Ceramic plates	
4.2	**-1500**	**M**	Ceramic plates	**Permanent Wilting Point (PWP)**
7.0	**-10^6**	**M**	**Oven**	**Dry Bulk Density (BD)**

So this ring test included 9 parameters of which six were mandatory and three were optional. All analyses had to be performed in 5 replicates.

2.7 Statistical data analysis

2.7.1 Between and within-laboratory variance

The aim of the statistical analysis is to answer the question "Which laboratories are performing well and which poorly?" based on the between-laboratory and the within-laboratory variance.

This analysis is using the international standard 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' (ISO, 1994). Data analysis was done by means of the statistical software package TIBCO Spotfire S^+ 8.1 for Windows (November 2008). This transparent and easily to interpret procedure adds some specific items to the classical procedure:

1. The interpretation of statistics has been facilitated by graphs integrating multiple statistical parameters.

2. The procedure is iterative. The presence of very deviant outliers can distort the view of the whole distribution. Multiple outliers can mask each other; by eliminating outliers, new outliers and stragglers may pop up. After outliers are eliminated, the statistical analysis is repeated to study the distributions in order to trace 'new' outliers or stragglers. This iterative procedure will continue until no new outliers are found or in this ring test, up to a maximum of three iterations in this interlaboratory comparison.

3. The procedure allows the comparison of different sources of variance:

$$sRepr^2 = sLab^2 + sRep^2$$

where $sRepr^2$ = estimation of the reproducibility variance

$sLab^2$ = estimation of the between-laboratory variance

$sRep^2$ = estimation of the repeatability (within-laboratory) variance

The reproducibility (Repr) is a measure of agreement between the results obtained with the same method or identical test or reference material under different conditions (execution by different persons, in different laboratories, with different equipment and at different times). The repeatability (Rep) is a measure of agreement between results obtained with the same method under the same conditions (job done by one person, in the same laboratory, with the same equipment, at the same time or within a short time interval). The between-laboratory variance is a measure of agreement between the results obtained with the same method or identical test or reference material in different laboratories.

2.7.2 Coefficients of variation (CV)

Based on the general mean (Mgen) and the reproducibility variance (sRepr), the coefficient of variation could be calculated. The coefficient of variation is defined as:

$$CV = \frac{\sigma}{\mu} \times 100 = \frac{s\,\mathrm{Re}\,pr}{Mgen} \times 100$$

Where σ = General standard deviation (estimated by the sRepr in the Mandels h/k plot)

μ = General mean (estimated by the Mgen in the Mandels h/k plot)

The CV provides an idea of the average deviation for a certain parameter. As the CV is standardised, it is possible to compare the CV's of the different parameters, and rank the analysed parameters according to their CV.

The CV is thus calculated based on the cleaned dataset after outliers have been removed. This CV includes both the within – and between laboratory variability which explains why the CV's in the FSCC ring tests are higher compared to ring tests where only the between-laboratory variability is evaluated. The proportion of the CV caused by the difference between the labs is given by the statistic 'Plab'. The proportion of the CV cause by the difference within the labs is given by 100 – PLab.

$$Plab = sLab^2/sRepr^2 * 100$$

3 Results and discussion

3.1 Participation

Twenty-seven laboratories registered to the ring test. Five of these labs do not participate in the FutMon D3 action. They all received their filled soil cores by the beginning of April 2009. Four laboratories received two set of cores: Lab F03 had one set of cores of 100 cc (F03a) and one set of 180 cc (F03b). Lab P03 and P14 had one set of 250 cc (P03a and P14a) and one set of 19 cc (P03b and P14b). The small cores were only used for the higher suction/pressure levels. Lab F15 received two sets of 100 cc to test two different methods (F15a and F15b).

The deadline for data submission was the 15th of August 2009. Since meeting this deadline was for several laboratories a problem, all data that reached FSCC by the 11th of September 2009 were included in the data analysis. By that time, 23 laboratories reported their results. Two withdrawn laboratories do not participate in the FutMon D3 action. The coordinates of the laboratories that reported results are given in Annex 1. Lab F03 and P14 reported data on both sets of rings that they received. Laboratory F15 reported on the second set of rings only the volumetric water content (VWC) at a pressure head which was not reported by any other lab, so this parameter could not be evaluated in this ring test.

Laboratory P10 reported one set of fitted values after measurements by the HYPROP instrument (UMS München) using a Van Genuchten bimodal model. The HYPROP instrument is based on the evaporation method according to Wind (1968) and Schindler (1980) which is applicable to measurements of the drying or desorption curve. The Wind's evaporation method is described by ISO 11275 (2004). The advantage is that the hydraulic properties are determined during a natural drying process of the soil.

Note that this is not a reference method of the FutMon protocol. The results have been included in the statistical data evaluation although one needs to keep in mind that the ring test compares in first instance between laboratories and has not been set up to compare methods.

Some laboratories measured the water content at (slightly) different pressure heads than requested but reported it as if it was measured at a requested pressure head. The results were analysed for the pressure heads as they has been reported by the laboratories.

In total 935 observations were reported. See Table 4. All results had to be reported in 5 replicates. Sometimes some data were missing or not yet available at the time of the data submission. As long as more than 1 replicate was reported, the results were included in the statistical analysis. The maximum number of data sets per parameter was 25 (dry BD). Most of the mandatory parameters were reported by all labs. Data in green colour cells were included in the statistical data evaluation (N= 930).

Table 4: Number of reported data by the 23 laboratories at the different pressure heads. Data in green colour cells were included in the statistical data evaluation (N= 930).

N° reported data	Parameter										
LabID	VWC-0	VWC-1	VWC-5	VWC-10	VWC-33	VWC-50	VWC-100	VWC-250	VWC-1500	dryBD	Total N° submitted results
Mand./Opt./ Not asked	M	M	M	O	M	Not asked	O	O	M	M	
A66	5	5	5	5	5		5		5	5	40
F03a	5	5	5	5	5		5	5	5	5	45
F03b	5	5	5	5	5		5	5	5	5	45
F10	5	5	5		5			5	5	5	35
F12	5	5	5	5	5		5	5	5	5	45
F15a		5	5		5					5	20
F15b						5					5
F17	5	5	5	5	5				5	5	35
F23	5	5	5		5				4	5	29
F27	5	5	5	5	5		5	5	5	5	45
P01	5	5	5	5	5				5	5	35
P02	5	5	5	5	5		5		3	5	38
P04	5	5	5	5	5		5		5	5	40
P05	5	5	5	5	5		5	5	5	5	45
P06	5	5	5	5	5		5	3	5	5	43
P08	5	5	5	5	5				5	5	35
P09	5	5	5	5	5		5		5	5	40
P10	4	4	4	4	4		4	4	4	4	36
P11	5	5	5	5	5		3	3	3	5	39
P12	5	5	5	5	5			5	5	5	40
P13	5	5	5	5	5		5		5	5	40
P14a	5	5	5						5	5	25
P14b					5		5	5	5	5	25
S01	5	5	5		5		5		5	5	35
S04	5	5	5		5				5	5	30
S22	5	5	5	5	5		5	5	5	5	45
Total N° data sets	23	24	24	18	24	1	16	12	24	25	935

3.2 Statistical data analysis

The data analysis using S-plus produced for each parameter a total of 7 figures: one dot plot of all reported values, one histogram and one box plot of the mean of the five reported replicates, one histogram and one box plot of the standard deviations, and one Mandel's h and one Mandel's k plot. All these graphs can be consulted in Annex 2.

3.2.1 Exploratory Data Analysis

The exploratory data analysis allows a visual evaluation of the data and gives an indication of possible outliers. However, based on these exploratory analysis, no observations nor laboratories have actually been excluded from further analysis.

Two sources of variance are investigated: the inter-laboratory variance (between-laboratory variance) and the intra-laboratory variance (within-laboratory variance). Figure 1 and Figure 2 represent the inter-laboratory variance. They indicate the position of each laboratory in the population of all laboratories. Figure 3 and Figure 4 represent the standard deviations of each laboratory. They yield information on the within-laboratory variance. Figure 1 and 3 are histograms, while Figure 2 and 4 are box-plots. The histograms provide a first rough overview of the distribution of all data reported for a certain parameter and sample. The information contained within the histograms is:

- Outliers that are 'very deviant' (parameter value and labID between parentheses)
- Relative frequencies in each class (in %)
- Density curve (smoothed trend-line)
- N: Number of observations in the histogram
- NA: Not Applicable

- Z: Number of reported zero's
- E: Number of excluded observations (very deviant outliers) from the presentation in the histogram; separately mentioned for upper and lower limits of distribution. The first number refers to the left side of the diagram, the second number to the right side of the histogram.
- U: Number of used observations in the calculations of a, m and s
- a: average value of the U observations
- m: median value of the U observations
- s: standard deviation of the U observations

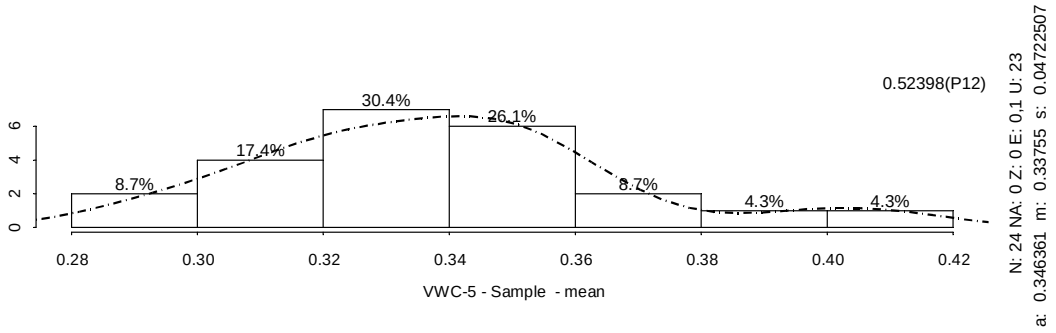

Figure 1: Histogram showing relative percentages and a rescaled density curve of the <u>mean</u> of five replicates of the measured parameter 'VWC-5' (volumetric water content at -5 kPa) by 24 laboratories. The units of the X-axis are in m^3 m^{-3}.

The information in the box plot starts from the dataset after the first rough cleaning done in the histograms where the 'very deviant' outliers have been excluded. The box plot provides following information:

- 'Visual' outliers (parameter value and lab N° between parentheses). These are placed in the top left and top right corner of the figure. On the right side of the figure 'O' indicates the number of outliers excluded from the box plot, respectively on the lower and the higher range of the box-plot. So in this example, seven outlying labs have been identified in the box plot on the lower range and four on the upper range.
- Percentiles Q1 (25%), Q2 (50% or median) and Q3 (75%)
- U: Number of observations in the box-plot where U=N-E in the histograms.
- Laboratories whose observations correspond to the median value, are put between brackets "< >"; observations between Q1 and Q2 are between "< <" and between Q2 and Q3 "> >".

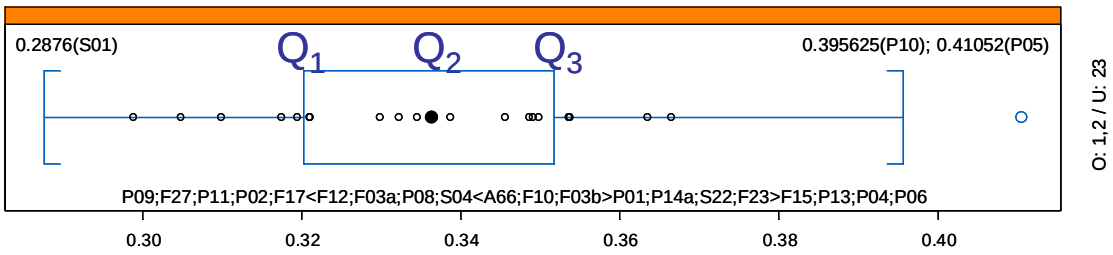

Figure 2: Box plot of the <u>mean</u> values reported for the volumetric water content at -5 kPa by 23 laboratories. The units of the X-axis are in m^3 m^{-3}.

Both histograms and box plots show the distribution after the 'very deviant' outliers have been excluded. 'Very deviant' outliers are located more then 3.5 times beyond the inter-quartile range (IQR). The IQR is defined as the distance from Q1 to Q3 (see Figure 2). In the box-plot the whiskers are placed at 1.5 * IQR. Observations outside the whiskers are the

'visual' outliers. It is possible that whiskers are placed on a closer distance than 1.5 * IQR in case there are no observations outside the 1.5 * IQR.

From the text on the right side of Figure 1, it can be observed that the histogram is based on results from N=24 laboratories. None of the reported values, was a "0" (Z: 0). One laboratory (P12) is excluded from the histogram, so the results of U= 23 laboratories are included in the calculation of the general statistics. Laboratory P12 reported extremely high volumetric water contents at pF 1.7 (-5 kPa), namely on average 0.5240 m^3 m^{-3} while the average water content is a: 0.3464 m^3 m^{-3} and the median is m: 0.3376 m^3 m^{-3} and standard deviation s: 0.0472 m^3 m^{-3}. In order to allow calculations of average, standard deviation and the Mandel's h and k statistics, data are supposed to have a normal distribution. The shape of the density curve (dotted line) should therefore approach the symmetrical shape of a normal distribution.

Figure 2 shows that the laboratories A66, F10 and F03b reported the median value of m: 0.3376 m^3 m^{-3}. Laboratories F12, F3a, P08 and S04 reported values between the first quartile (Q1) and the median (m); laboratories P01, P14a, S22 and F23 reported values between the median and the third quartile (Q3). Laboratories P09, F27, P11, P02 and F17 reported values below the first quartile (Q1) and laboratories F15, P13, P04 and P06 reported values above the third quartile (Q3). The laboratories outside the 1.5 * IQR whiskers are given with their laboratory number and average value above the box plot. Laboratories S01 reported very low VWC of 0.2876 at -5 kPa and Labs P10 and P05 reported very high VWC at -5 kPa of more than 0.3956 m^3 m^{-3}.

Based on the histogram of the means (Figure 1), one would expect that laboratory P12 will be an outlier in the in-depth statistical analysis for the between laboratory variability. Based on the box plot which is more severe (Figure 2), we see that laboratories S01, P10 and P05 also have doubtful results.

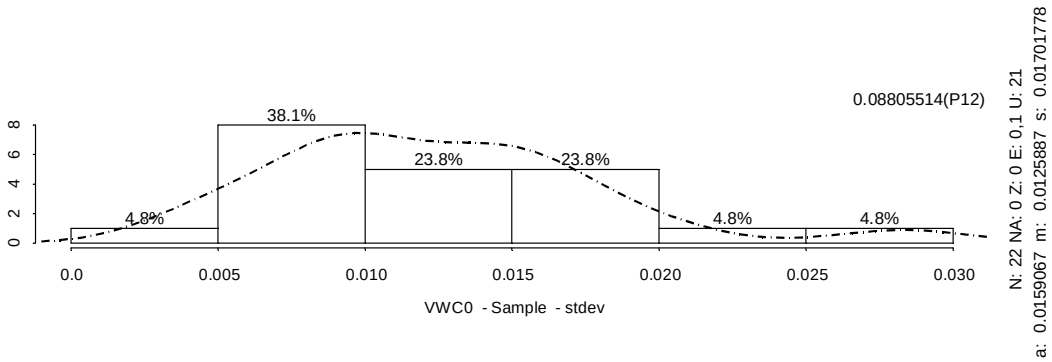

Figure 3: Histogram showing relative percentages and a rescaled density curve of the <u>standard deviations</u> of five replicates of the measured VWC at 0 kPa by 22 laboratories. The units of the X-axis are in m^3 m^{-3}.

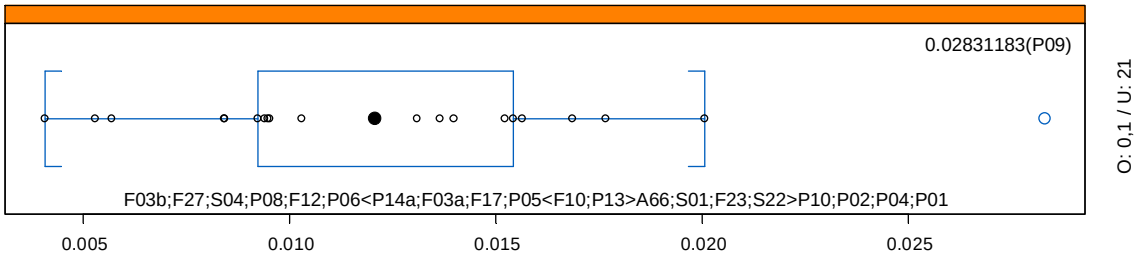

Figure 4: Box plot of the <u>standard deviations</u> of five replicates of the measured VWC at 0 kPa by 21 laboratories. The units of the X-axis are in m^3 m^{-3}.

The histogram of the standard deviations (Figure 3) shows one very deviant outlier (P12). The more severe box plots, defines one visual outlier (P09) for the within-laboratory variability.

A laboratory can also check its performance compared to the other laboratories by studying the dot plot (Figure 5). Every dot represents a reported value of a specific parameter. The shape of the dot plot follows the sigmoid curve shape of a normal distribution. Laboratories are plotted on the Y-axis, arranged according to the magnitude of the reported values. One laboratory reported extremely deviant results for the VWC at 0 kPa. Two of the five values are given at the top of the graph in order not to distort the figure too much. Values reported by other laboratories can be read on the X-axis.

Such a figure can also tell something about the internal variance within one laboratory. For example, laboratories F03b reported five very similar results – represented by 5 dots close to each other – whereas laboratories P09 reported 5 very different results – represented by 5 dots widely separated from each other. We expect that laboratory F03b will have a good within-laboratory repeatability whereas laboratory P09 will have a very poor within-laboratory repeatability.

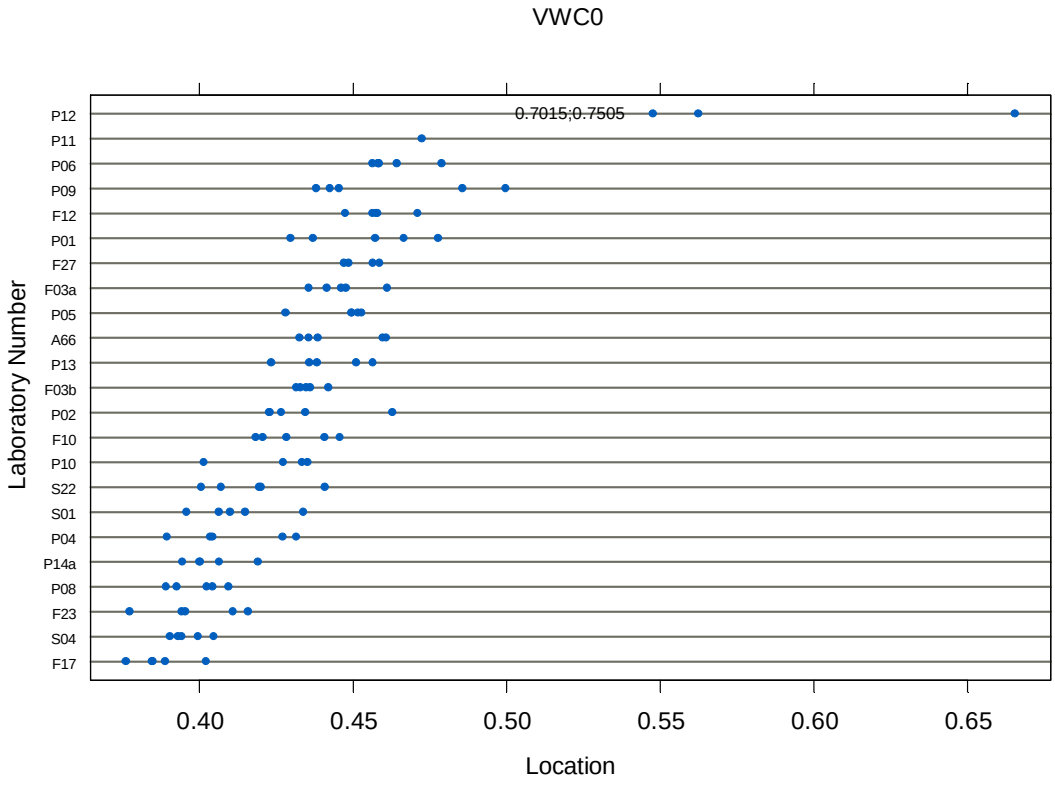

Figure 5: Dot plot of reported VWC at 0 kPa, ordered increasingly

3.2.2 In-depth statistical data analysis: Mandel's h and k statistics

Figure 6 presents an example of the Mandel's h and k plot for the VWC at 0 kPa. The Mandel's h statistic tests the between-laboratory variance. The Mandel's k statistic is a measure for the within-laboratory variance. The information contained within the two figures is:

- Step x: Iteration number of runs; varies in this interlaboratory comparison from 1 till maximum 4 steps in the VWC at – 1500 kPa
- Nlab: Number of laboratories after elimination of outliers
- Mgen: General mean after outliers have been excluded

- Fval: tests whether interlaboratory variance $\sigma_L^2 \neq 0$, F test for laboratory effect
- Pval: tests whether interlaboratory variance $\sigma_L^2 \neq 0$, p value of the F test
- $sRep^2$: estimation of repeatability variance
- $sLab^2$: estimation of the between-laboratory variance
- $sRepr^2$: estimation of the reproducibility variance
- CV: coefficient of variation (σ/μ)*100 = sRepr/Mgen*100
- Excluded laboratories: excluded observations that are statistical outliers, mentioning whether it was based on the h or k statistic:
- "h (H) + Laboratory N°": laboratory has been excluded based on the Mandel's h statistics
- "k (K) + Laboratory N°": laboratory which has been excluded based on the Mandel's k statistics
- E: Excluded observations, mentioning whether it was based on the h or k statistics

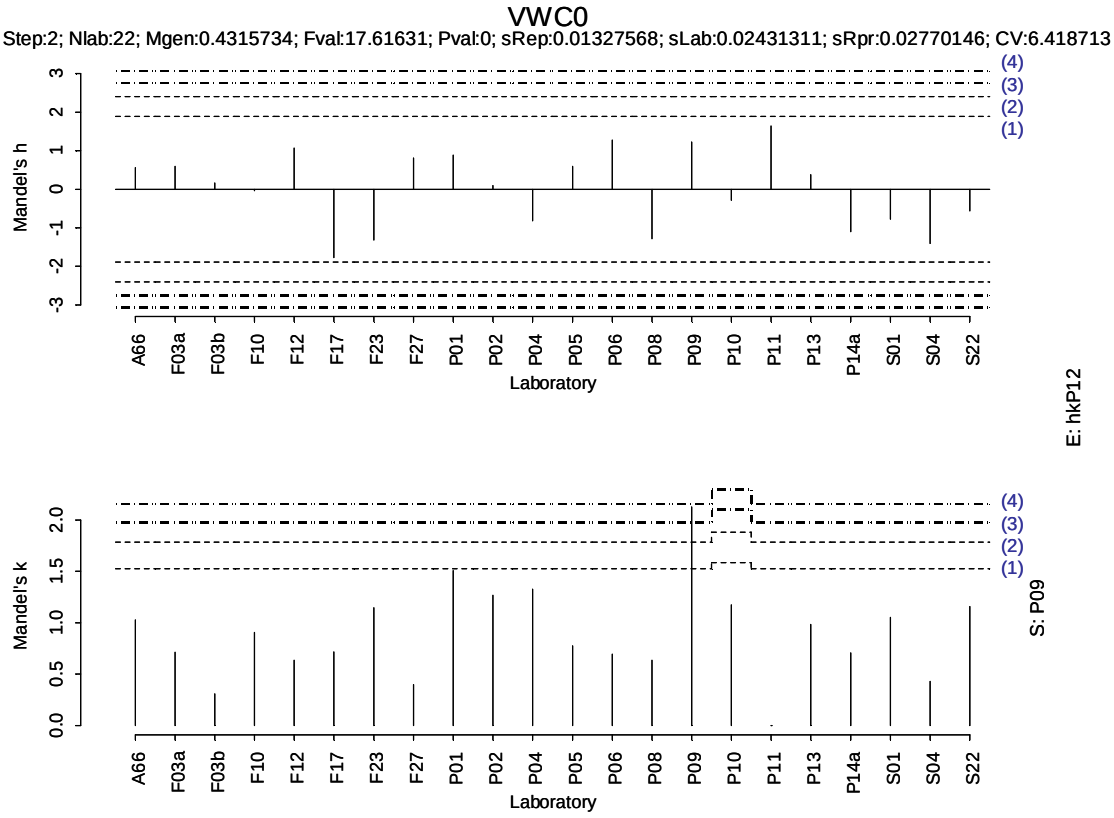

Figure 6: Mandel's h and k statistic for the VWC at 0 kPa

On both the Mandel's h and k plots, 4 critical levels are indicated. When the critical level is exceeded, the H-null hypothesis "no difference between the mean values" will be rejected.

(1) Critical value where H_0 will be rejected at probability level of 95%
(2) Critical value where H_0 will be rejected at probability level of 99%
(3) Critical value where H_0 will be rejected at probability level of 95% after application of the Bonferroni rule.
(4) Critical value where H_0 will be rejected at probability level of 99% after application of the Bonferroni rule.

Statistical outliers are the observations of which the Mandel's h or k-statistic exceeds the critical value at probability level of 99% after application of the Bonferroni rule. Statistical stragglers are the observations of which the h or k-statistic are situated between the critical values of probability level 95 and 99% after application of the Bonferroni-rule. Figure 6 forms the core of the statistical analysis and contains all necessary information. It usually confirms the expectations after studying Figures 1 till 5.

The Mandel's k statistic of laboratory P09 is high, but does not reach critical limit N°4 (Figure 6). It is a straggler because the Mandel's k value is located between the critical value of the 95% and 99% confidence limits, and identified as such on the right side of the figure by the letter 'S' followed by the labID. It confirms what was already shown by Figure 4.
Laboratory P12 has been excluded from the statistical analysis based on the Mandel's h and based on the Mandel's k statistics (see right side of Figure 6 'E' followed by a small letter 'h' and 'k'). When looking at the histogram of the means in Annex 2, and at Figure 3 above, the lab was indeed identified as a very deviant outlier based on the within- and the between-laboratory variability.

When either the Mandel's h or k value is located between the critical levels (2) and (3), the laboratory results are located in the tail of the distribution. This is for example the case for Lab P13 for the VWC at -5 kPa in Figure 7.

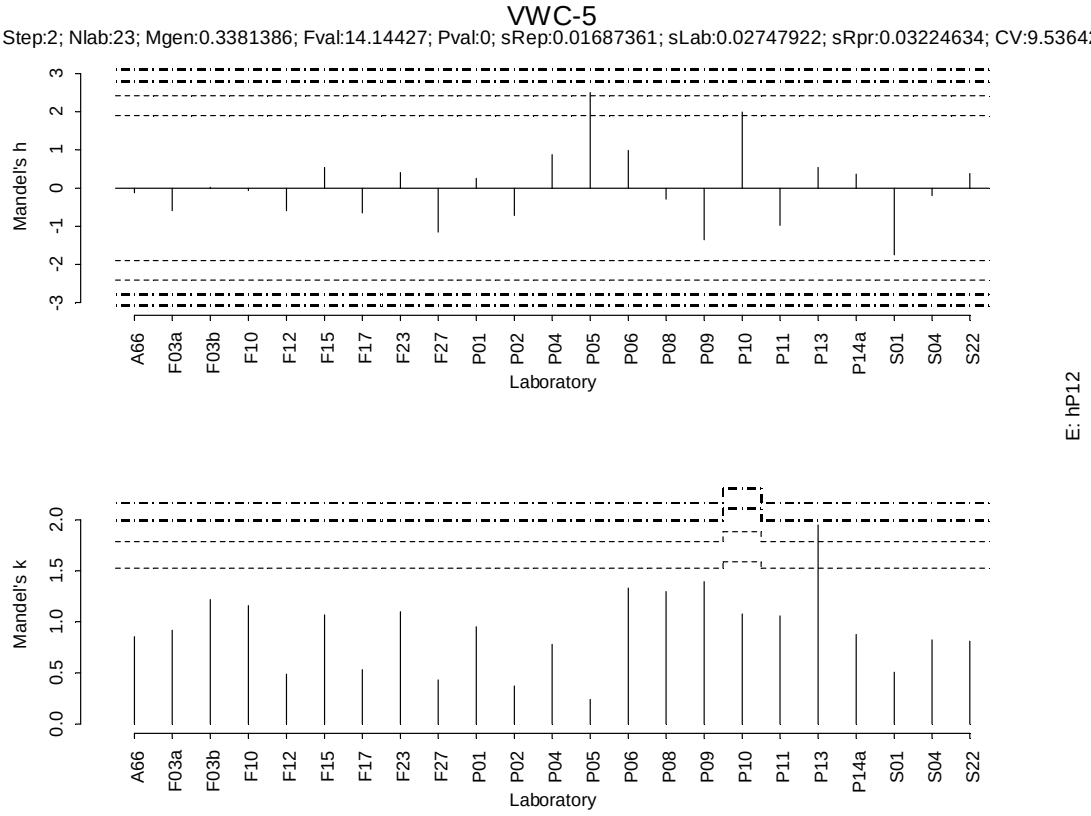

Figure 7: Mandel's h and k statistic for the VWC at -5 kPa

The critical level in the Mandel's k plot for the lab P10 has been adjusted. Since the laboratory reported only 4 replicates, the degrees of freedom in the calculation of the critical levels will be less and so the confidence interval will be wider, as shown in Figure 7.

3.2.3 The outlier free mean and the coefficient of variation

After the iterative procedure of outlier detection, the general cleaned mean and the related coefficient of variation has been calculated (Table 5).

Table 5: The general mean and coefficient of variation without and with exclusion of the outliers

Parameter	Units	M/O	Tot N° results	N° Labs	General mean	CV	Excluded labs	Step	Tot N° results	N° Labs	General cleaned mean	CV
VWC0	$m^3.m^{-3}$	M	114	23	0.44	12.45	hkP12	2	109	22	0.43	6.42
VWC-1	$m^3.m^{-3}$	M	119	24	0.39	13.13	hP12	2	114	23	0.38	7.46
VWC-5	$m^3.m^{-3}$	M	119	24	0.35	14.33	hP12	2	114	23	0.34	9.54
VWC-10	$m^3.m^{-3}$	O	89	18	0.30	10.57	hP12	2	84	17	0.30	8.18
VWC-33	$m^3.m^{-3}$	M	119	24	0.25	11.49	hP12	2	114	23	0.25	8.68
VWC-100	$m^3.m^{-3}$	O	77	16	0.20	10.53	kP11;kP02	3	69	14	0.20	10.03
VWC-250	$m^3.m^{-3}$	O	55	12	0.15	19.11	kP11	2	52	11	0.15	19.12
VWC-1500	$m^3.m^{-3}$	M	114	24	0.11	41.22	kP11;kA66	3	106	22	0.11	42.32
dryBD	$kg.m^{-3}$	M	124	25	1431	4.53		1	124	25	1431	4.53

Laboratory P12 has been excluded for five of its eight reported measurements based on its between-laboratory variability. It is clear that this laboratory will be asked to solve its problems before reporting results to the central FutMon database.

Laboratory P11 shows three outliers (VWC at -100, -250 and -1500 kPa) based on its within-laboratory variability. This is due to one outlier on its three reported values which was already noticed by the laboratory itself before reporting. This can clearly be observed from the dot plots and the box plots of the standard deviations in Annex 2. The laboratory reported only three replicates for these three pressure heads because they did not have sufficient material for the analysis on the pressure plate of the disturbed soil material. This can easily be solved during a survey by providing more loose soil material. When more replicates are available, the elimination of one outlier will be less problematic. So no immediate follow up actions for this laboratory are required.

Laboratory P02 was excluded for the VWC at – 100 kPa based on its poor within laboratory variability which was due to one outlier. Laboratory A66 was excluded for the VWC at – 1500 kPa based on its within laboratory variability. As for lab F11, we do not consider this as a major problem.

The exclusion of the outliers influences the general mean but not drastically. Most of the coefficients of variations dropped after the exclusion of the outliers, especially when there were outliers detected for the between laboratory variability. The magnitude of the CVs, is compared to our experience in the chemical soil ring tests, very good. Six of the nine parameters show a CV lower than 10%. The most difficult parameters to analyse were the VWC at the higher pressure heads of – 250 and -1500 kPa. The VWC at – 1500 kPa had a CV of 42% after the exclusion of two laboratories with a too high within laboratory variability. The high coefficient of variation can mainly be explained by the high number of tail values in the normal distribution (see Figures 54 and 55 in Annex).

The coefficient of variation on the dry bulk density is less than 5% which is very low. This result was however obtained after a first validation by FSCC checking the plausibility range of dry bulk density of European mineral forest soil between 890 and 1570 kg m^{-3} (interquartile range of the data of the Forest Soil Condition database).Three laboratories were outside this range. FSCC asked them to check their data. The tree labs could easily trace back their calculation and/or reporting mistake and so could resubmit their corrected values.

The coefficients of variation mentioned in Table 5 are a combination of the between and within laboratory variation. Table 6 shows the relative contribution of the within- and between-laboratory variance to the total variance.

Table 6: The relative distribution of the CV due to the within – and the between-laboratory variance

Parameter	CV(%)	% Between lab variance	% Within lab variance
VWC0	6.4	77	23
VWC-1	7.5	78	22
VWC-5	9.5	73	27
VWC-10	8.2	59	41
VWC-33	8.7	80	20
VWC-100	10.0	83	17
VWC-250	19.1	90	10
VWC-1500	42.3	98	2
dryBD	4.5	36	64

In line with the expectations, for most of the parameters the variance is caused by differences between the labs. This is however not the case for the dry bulk density. When most of the variance is due to difference between the labs, further harmonisation efforts between the laboratories should improve the variability of the results. So especially at the high pressure heads measured on the pressure membrane cells and on the pressure plate extractors of – 250 kPa and at -1500 kPa further harmonisation is required.

3.2.4 Ranking of the laboratories

Figure 8 presents a ranking of the laboratories based on the number of identified outliers, stragglers and tail values (at 95% and 99% confidence levels) in the statistical data analysis. Tail values are no exceptional values but are located in the tail of the normal distribution. Equal weight has been given to the within – and the between-laboratory variance. The Figure presents the absolute number of outliers, stragglers and tail values so laboratories that reported all optional and mandatory parameters have more chance to pop up in this figure.

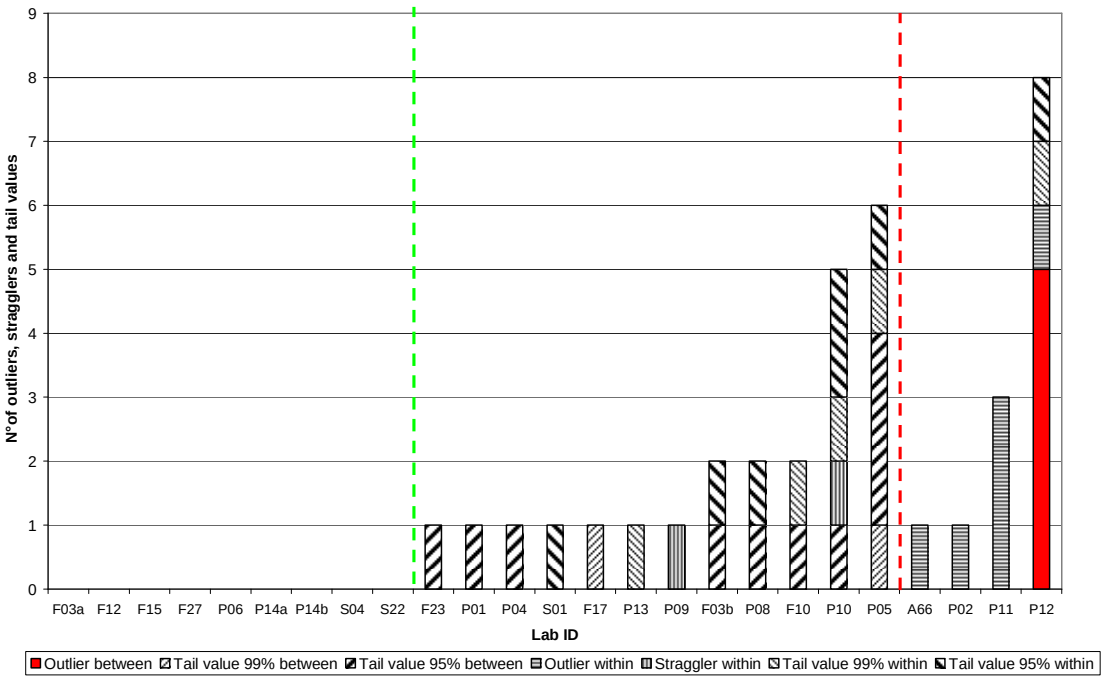

Figure 8: Number of reported outliers, stragglers and tail values by the 23 laboratories for both the within – and between-laboratory variability

The eight laboratories (F03a, F12, F15, F27, P06, P14, S04 and S22) on the left reported always values which were nicely within the bulk of the data. The four laboratories which were excluded for at least one parameters are shown on the right side of the figure (A66, P02, P11 and P12). Of the laboratories in the central part of the x-axis, P09 and P10 had one straggler for the within laboratory variance. For all the other laboratories in the centre the height of

the bar is a measure for the number of reported tail values. So although the laboratories P05 and P10 did not show any outliers, their results are often located in the tail of the normal distribution.

The laboratory P10 that used the HYPROP technique showed one straggler (so an outlier at the 95% confidence interval) and three tail values for the within-laboratory repeatability and one tail value for the between-laboratory variability.

3.2.5 Feedback from the participants

At the 2nd Meeting of the Heads of the Laboratories (Warsaw, 12-13 October 2009) the results of the ring test were presented and discussed.

Laboratory P12 found an error in the calculation procedure of it volumetric water contents and provided FSCC the corrected results. Its results for the measurements at -10kPa and -250 kPa were further removed from the data set as these optional parameters were measured at different matric pressures.

Although no qualification/requalification procedure is foreseen in this ring test, the general statistics were calculated again after the correction made by lab P12. See Table 7.

Table 7: The outlier free mean and coefficient of variation without and with exclusion of the outliers after correction by Lab P12.

Parameter	Units	M/O	Tot N° results	N°labs	General cleaned mean	CV	Exluded labs	Tot N° results	N°labs	General cleaned mean	CV
VWC0	m^3m^{-3}	M	114	23	0.4298	6.98	kP12	109	22	0.4316	6.42
VWC-1	m^3m^{-3}	M	119	24	0.3787	7.44		119	24	0.3787	7.44
VWC-5	m^3m^{-3}	M	119	24	0.3373	9.44		119	24	0.3373	9.44
VWC-10	m^3m^{-3}	O	84	17	0.2992	8.18		84	17	0.2992	8.18
VWC-33	m^3m^{-3}	M	119	24	0.2450	9.17		119	24	0.2450	9.17
VWC-100	m^3m^{-3}	O	77	16	0.1969	10.53	kP11;kP02	69	14	0.1974	10.03
VWC-250	m^3m^{-3}	O	50	11	0.1524	17.24	kP11	47	10	0.1533	17.04
VWC-1500	m^3m^{-3}	M	114	24	0.1056	42.93	kP11;kA66	106	22	0.1057	44.16
dryBD	kg m^{-3}	M	124	25	1431	4.53		124	25	1431	4.53

After correction, Lab P12 found all its results for the between laboratory variability within the expected normal distribution curve. Yet, the values for the volumetric water content at saturation were still excluded based on the high variation between the replicates.

3.2.6 The soil water retention curve

Figure 9 compares the soil water retention curve of the mean volumetric water contents of the artificial sample with a natural sample taken from a similar horizon of the same site. Note however, that the dry soil bulk density of the artificial sample (1431 kg m^{-3}, SD = 65 kg m^{-3}) was higher than the bulk density of the natural sample (1301 kg m^{-3}, SD = 48 kg m^{-3}).

The soil water retention curve of the artificial ring test sample is similar to the SWRC of the natural undisturbed samples taken in the same horizon for the pressure heads below 3.0. The moisture content at the higher pF values of 3.4 and 4.2 are slightly higher compared to the natural sample. Hence, the artificially prepared samples compare well with field samples.

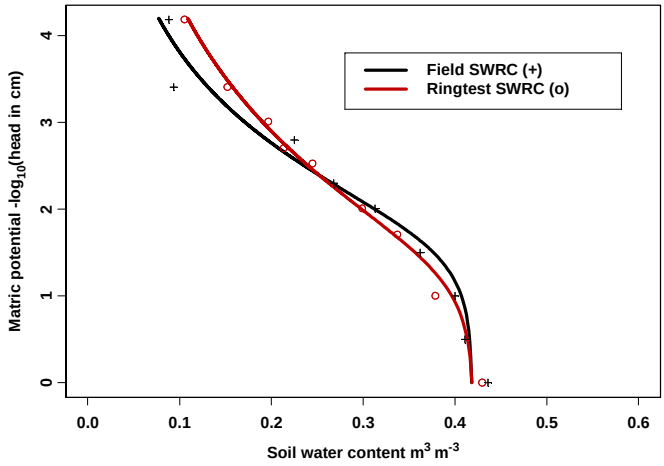

Figure 9: The soil moisture retention curve of the artificial ring test sample and the original undisturbed sample.

The mean soil water retention curve of each individual laboratory was compared with the general mean soil moisture curve of all the labs. Based on the Mualem - Van Genuchten equation (Van Genuchten, 1980) the four parameters describing the soil water retention curve were estimated (Figure 10).

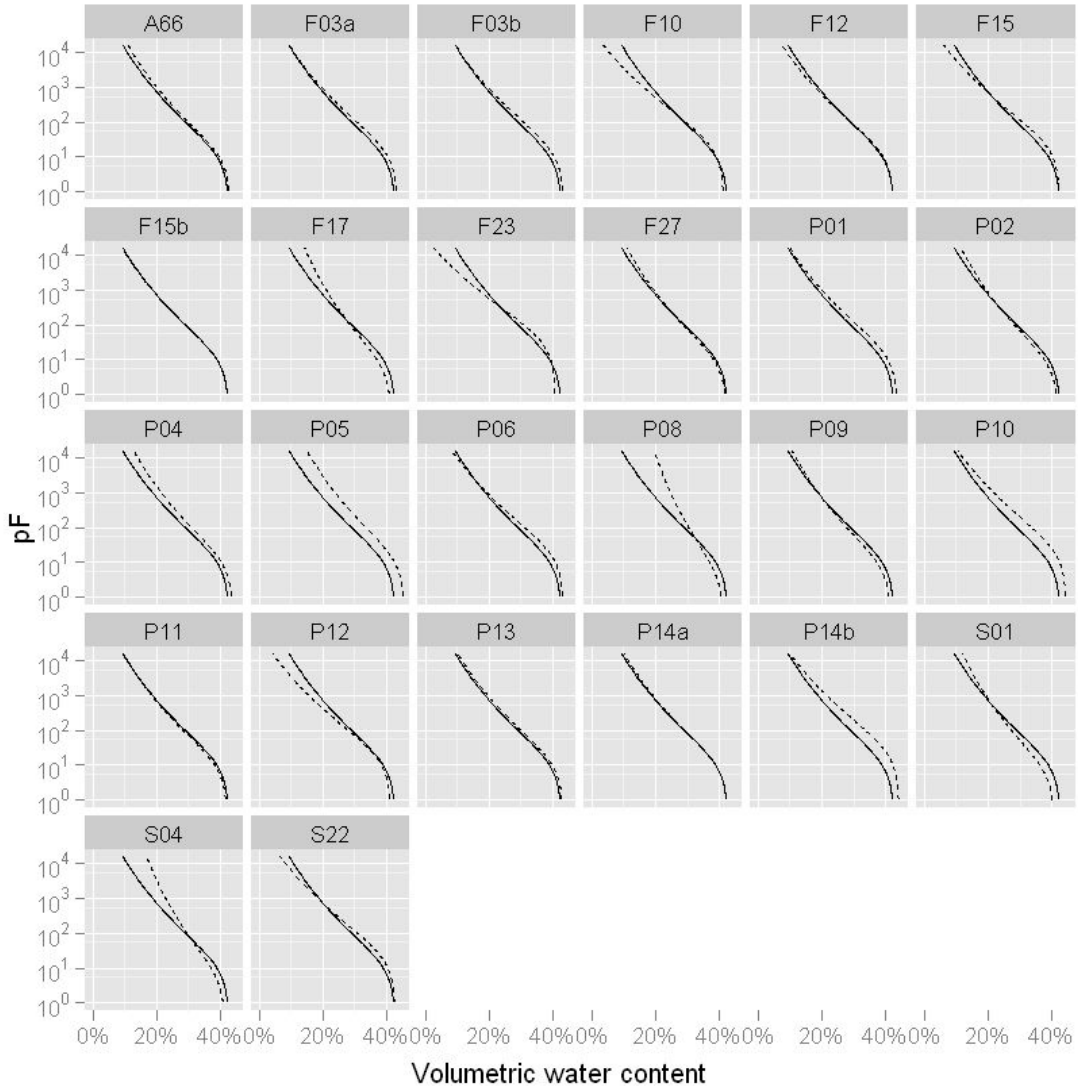

Figure 10: Comparison of the modelled soil water retention curves of the individual laboratories (dashed line) with the mean soil moisture retention curve (full line).

By studying the soil water retention curves, five outlying laboratories could be identified. The results reported by Laboratory P05 deviate strongly from the general mean soil water retention curve: at all matric pressures too high soil moisture values are estimated. At the high matric pressures Laboratories P08 and S04 deviate by estimating too high soil moisture contents and laboratories F10 and F23 by too low moisture contents.

Four other laboratories could be considered as stragglers. Laboratories P10 and P14b overestimate the soil moisture content at all matric heads. Laboratory P12 underestimates the soil moisture content at the high matric heads. Laboratories F17 and S01 underestimate the soil moisture content at low pressure heads and overestimate it at the high pressure heads.

3.2.7 Laboratory methods and equipment

Figure 11 illustrates the methods as reported during the data submission for this ring test.

pF 0.0 or 0 kPa

The volumetric water content at pF 0.0 or 0 kPa is equal to the total porosity. By weighing the saturated sample on an electronic balance – done by the majority of the labs-, there will always be some loss of water causing errors. Alternatively, the total porosity could be calculated based on the bulk density of the soil and the particle density or the true density of the soil. The latter is measured by a pycnometer. Five laboratories made this measurement. Three laboratories estimated the particle density based on the mineral density of quartz 2650 $kg.m^{-3}$ and/or the density of the organic material. Since the overall coefficient of variation (after the removal of one outlier) is not more than 6.42 %, the three methods of the protocol to determine the volumetric water content at pF 0.0 or 0 kPa should be accepted.

pF 1.0 or – 1 kPa: Sand suction table recommended

The protocol recommends the use of the sand suction table. Twelve laboratories made indeed use of the conventional sand tables and two laboratories used (self-made) ceramic suction tables. One laboratory used the pF laboratory station. One laboratory (F17) used the Buchner funnel, a method which is not described in the FutMon protocol but accepted by ISO.

A total of six laboratories measured the volumetric water content at matric potential of – 1 kPa using the ceramic plate extractor while this instrument is not suitable for this pressure head. Five of these laboratories use the equipment of Soilmoisture corp. which technically allows soil moisture measurements at pressures between 0 and 15 bar. On the other hand, the manual of this instrument recommends not to use this apparatus below 10 kPa because of the destruction of the soil structure which plays an important role in the volumetric water content at low (absolute) pressure heads.

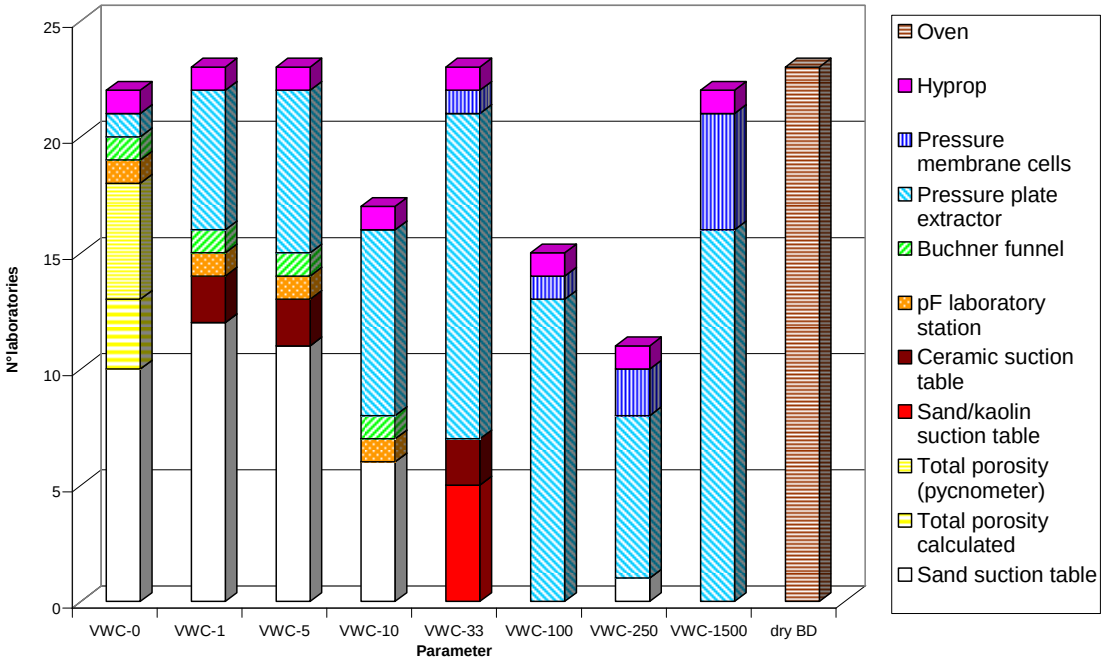

Figure 11: Reported laboratory methods for determination at each matric head

pF 1.7 or - 5 kPa: Sand suction table recommended

At pF 1.7 the same comments are valid as above for the pF 1.0. In this case eleven labs made use of the conventional sand suction table and seven laboratories used the ceramic pressure plate extractor.

pF 2.0 or – 10 kPa: Sand suction table recommended

At pF 2.0 the same comments are valid as above for the pF 1.0. In this case only six labs of the 17 labs that reported this parameter, made use of the conventional sand suction table and eight laboratories used the ceramic pressure plate extractor.

pF 2.5 or – 33 kPa: Sand/kaolin suction table recommended

At the matric potential of – 33 kPa, the protocol version 1.2. recommends the sand/kaolin suction table. One reason is that structure is a quite important factor in the value of -10 and – 33 kPa. So instruments that use a disturbed sample are less suitable for these measurements. However in the ring test we see that 14 out of 23 laboratories use pressure plates, while only 5 laboratories use the recommended instrument. One laboratory used the Ecotech pF laboratory station, one laboratory a ceramic plate with prevention for evaporation and one laboratory used the Hyprop instrument. One laboratory even used a pressure membrane apparatus (P08) while the FutMon protocol states, following ISO, that this method should only be used for pressures below – 33 kPa.

pF 3.0 or – 100 kPa: ceramic plates or pressure membrane recommended

Of the 15 laboratories that reported this optional volumetric water content, one laboratory used the pressure membrane method, one the Hyprop instrument and 13 the pressure plate extractor.

pF 3.4 or – 250 kPa: ceramic plates or pressure membrane recommended

Of the 11 laboratories that reported this optional volumetric water content, two laboratories used the pressure membrane method, one the Hyprop instrument and 7 the pressure plate extractor. One laboratory used the sand suction table.

pF 4.2 or – 1500 kPa: ceramic plates or pressure membrane

Of the 22 laboratories that reported this mandatory volumetric water content, five laboratories used the pressure membrane method, one the Hyprop instrument and 16 the pressure plate extractor.

pF 7.0 or - 10^6 kPa: recommended method: oven at 105°C

All laboratories dried the samples at 105°C in the oven to determine the dry bulk density.

3.2.8 Recommendations towards SA14 FutMon protocol

The FutMon protocol SA14 version 1.2 is not correct concerning the recommended method for the VWC at – 100 kPa. While Table 2 in the protocol recommends the kaolin suction table, the commercial available sand/kaolin suction tables can only be used up till – 50 kPa, conform the ISO 11274 (ISO, 1998) standard. On the other hand the ceramic pressure plate extractors can be used for pressures below -33 kPa (starting from – 5 kPa according to ISO11274) up to -1500 kPa. Pressure membranes are more suitable for pressures below -100 kPa.

Except for the Hyprop measurements, all the measurements at -100 kPa in this ring test were done using pressure plate extractors or a pressure membrane apparatus. The table in the FutMon protocol needs to be corrected (Table 8).

Table 8: Suggestion for adjusted table in the FutMon SA14 protocol

Matric potential ψ			Recommended instrument/Method	Estimator	M/O
cm H_2O	pF	kPa			
1	0.0	0	Pycnometer	≈θsat=WHC= Total porosity	M
10	1.0	-1	Sand suction table (method A)		M
51	1.7	-5			M
102	2.0	-10		FC sand	O
337	2.5	-33	Kaolin suction table (method A)	FC siltloam	M
1022	3.0	-100	Pressure plate extractor (method C) or Pressure membrane cells (method D)	FC clay	O
2555	3.4	-250			O
15330	4.2	-1500		PWP	M
10^7	7.0	-10^6	Oven	Dry BD	M

Where:
1) the pF is the logarithm of the absolute value of the matric potential expressed by the graduation of the water column (cm).
2) 1 kPa = 10.22 cm H_2O or 1 cm H_2O column = 0.097885 kPa
3) 100 kPa = 1 bar

4 Conclusions

Twenty-three laboratories participated in this first FSCC Soil Physical Ring Test. Based on the ISO methodology for the determination of repeatability and reproducibility of a laboratory measurement method four laboratories (A66, P02, P11 and P12) were excluded for at least one of the nine evaluated parameters. Eight laboratories (F03a, F12, F15, F27, P06, P14, S04 and S22) passed easily the ring test without any outlier, straggler or tail value for any of their reported parameters.

One laboratory (P12) was excluded based on poor between laboratory reproducibility for five of its reported parameters. Laboratories P11, P02 and A66 showed poor repeatability for some parameters because of one deviating subsample.

In all, the results of this ring test are very good. Six of the nine reported parameters show a coefficient of variation below 10%. Especially at the smaller matric potentials, we do not expect a considerable quality improvement when all the analyses would be performed by one central laboratory. The problems are mainly situated in the measurement of the volumetric water content at pressures of -250 and -1500 kPa. Since the latter is a critical value for estimating the available water capacity of soils, improvement in the interlaboratory comparability at the higher matric potentials remains an important challenge.

Concerning the methods, it needs to be clarified whether the ceramic pressure plate extractors can be used for pressure heads -1, -5, -10 and -33 kPa as this seems to be a common practice while it is not accepted by the present FutMon field protocol. It is beyond all discussion, that the volumetric water content needs to be reported at fixed pressure heads in order to come to a harmonised forest soil water retention curve database at European level.

Since the SWR results of the artificially prepared test samples compare well with undisturbed field samples taken from the same site, the applied methodology holds promise for organising other 'controlled' physical ringtests in the future. In contrast with this ringtest, a

wide range of (forest) soil samples with variable texture, coarse fragment and organic matter content could then be tested to evaluate inter- and intralaboratory performance.

5 Acknowledgements

We thank Mathieu Pieters, Koen Vervaet and Koen Willems for taking and preparing the ring test samples and the laboratory personnel of the INBO analytical laboratory, Ilse Temmerman, under the supervision of Els Mencke and Gerrit Genouw for conducting the homogeneity tests. Further we express thanks to the Institute's unit of Biometry, Methodology and Quality Assurance (BMK), more specifically to Pieter Verschelde for the statistical support.

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Volumetric water content at 0 kPa (pF 0.0)

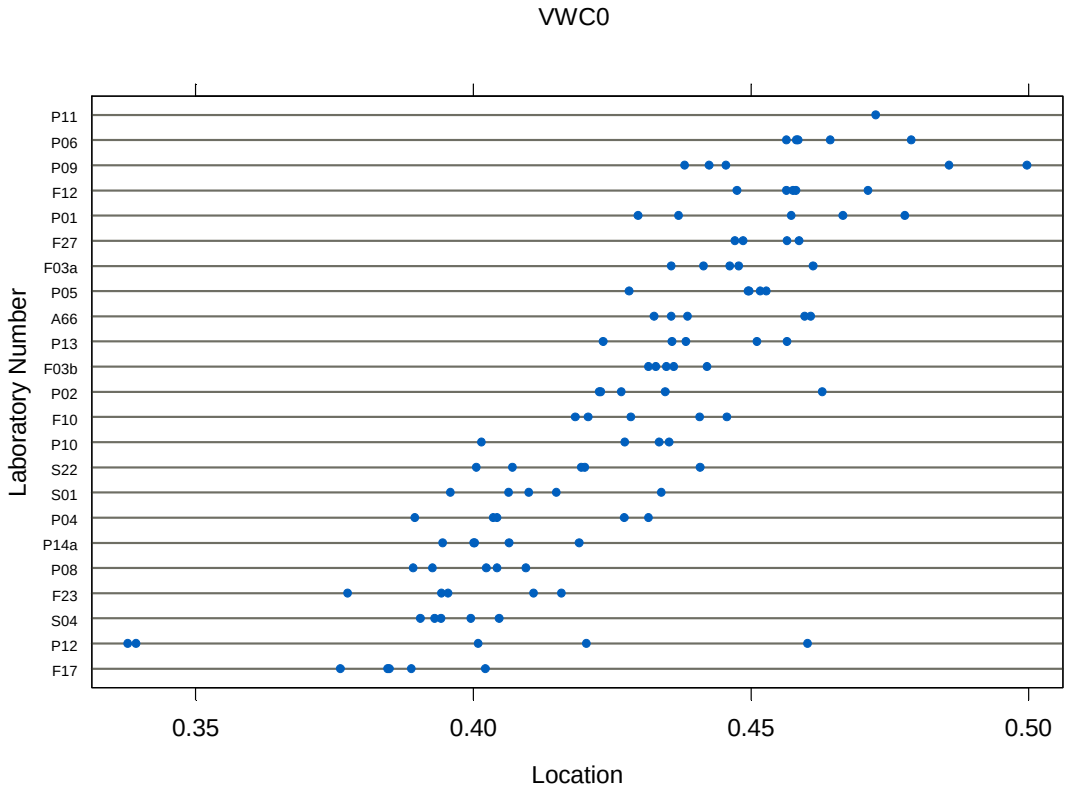

Figure 12: Dot plot of the volumetric soil water content at 0 kPa (pF 0.0) (m^3m^{-3})

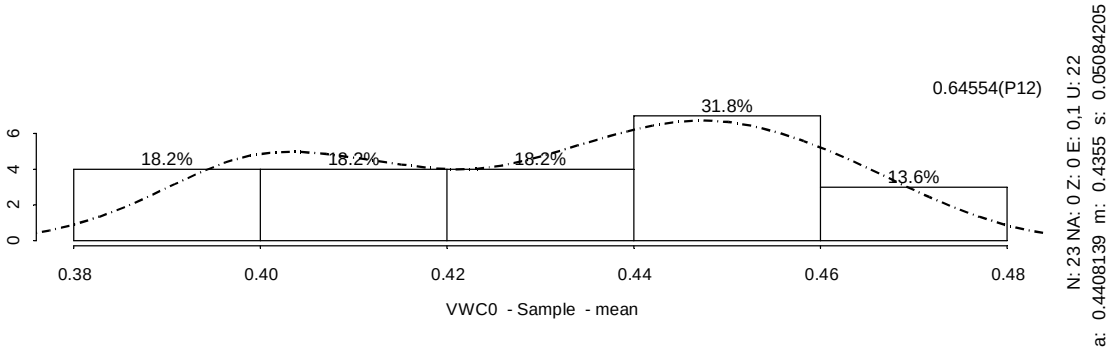

Figure 13: Histogram of the means of the volumetric soil water content at 0 kPa (pF 0.0) (m^3m^{-3})

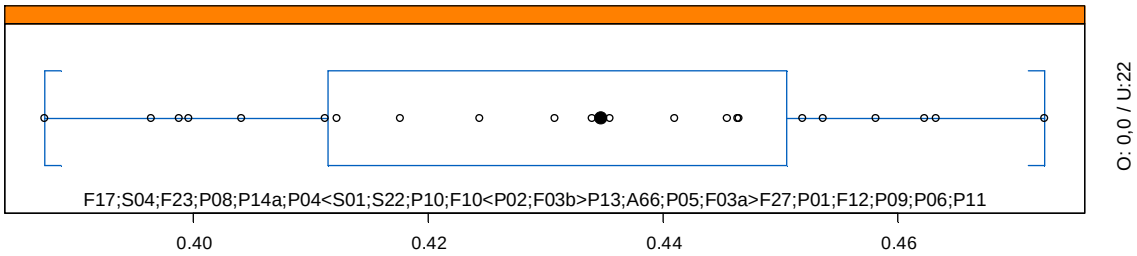

Figure 14: Box plot of the means of the volumetric soil water content at 0 kPa (pF 0.0) (m^3m^{-3})

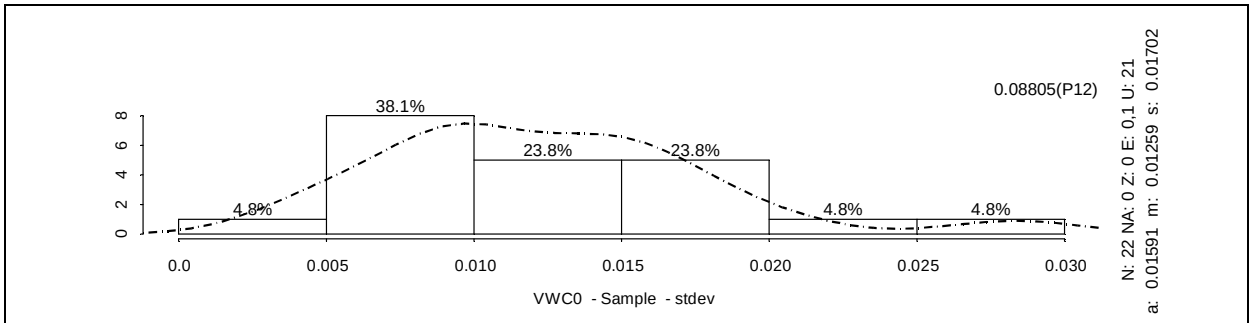

Figure 15 : Histogram of the standard deviations of the volumetric soil water content at 0 kPa (pF 0.0) (m^3m^{-3})

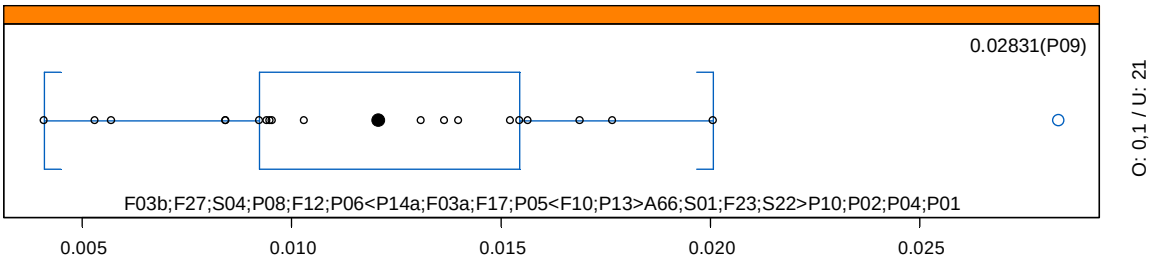

Figure 16: Figure: Box plot of the standard deviations of the volumetric soil water content at 0 kPa (pF 0.0) (m^3m^{-3})

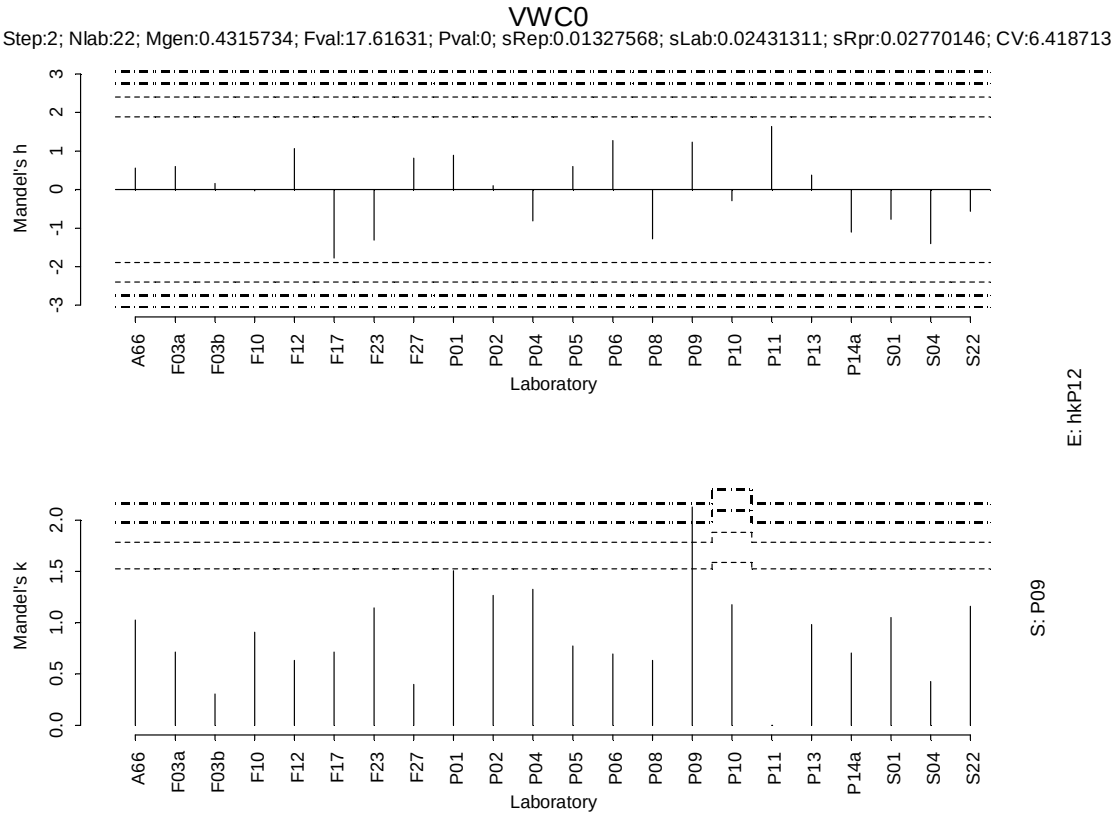

Figure 17: Mandel's h and k plot of the volumetric soil water content at 0 kPa (pF 0.0) (m^3m^{-3})

Volumetric water content at -1 kPa (pF 1.0)

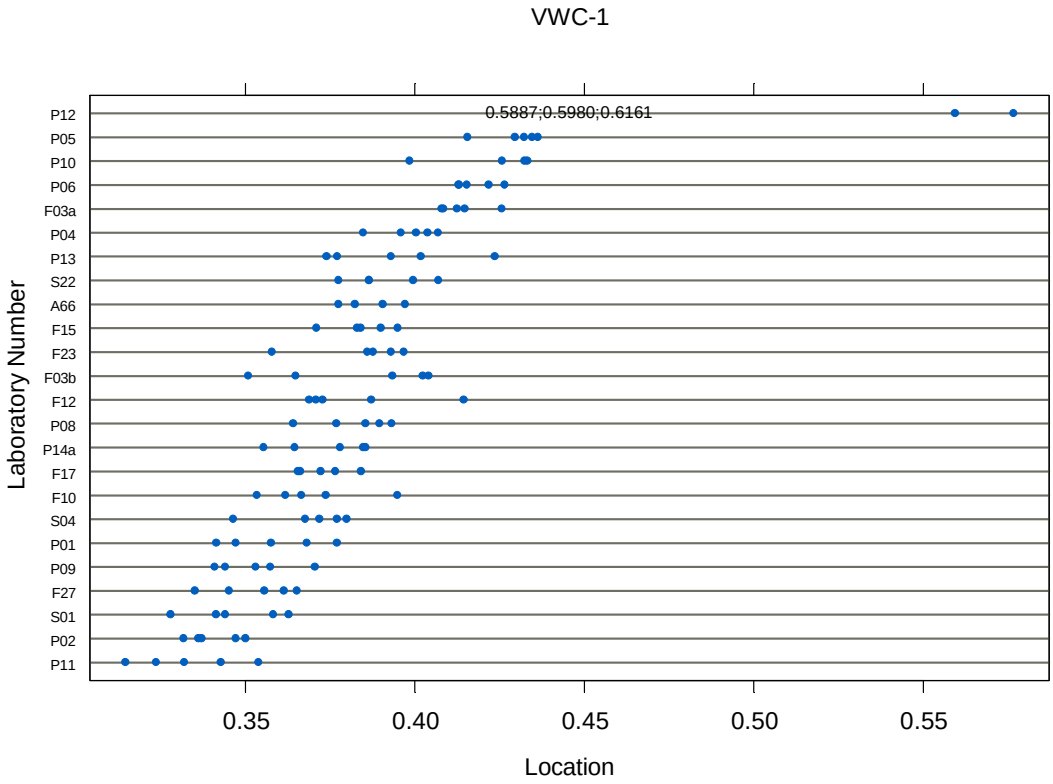

Figure 18: : Dot plot of the volumetric soil water content at -1 kPa (pF 1.0) (m^3m^{-3})

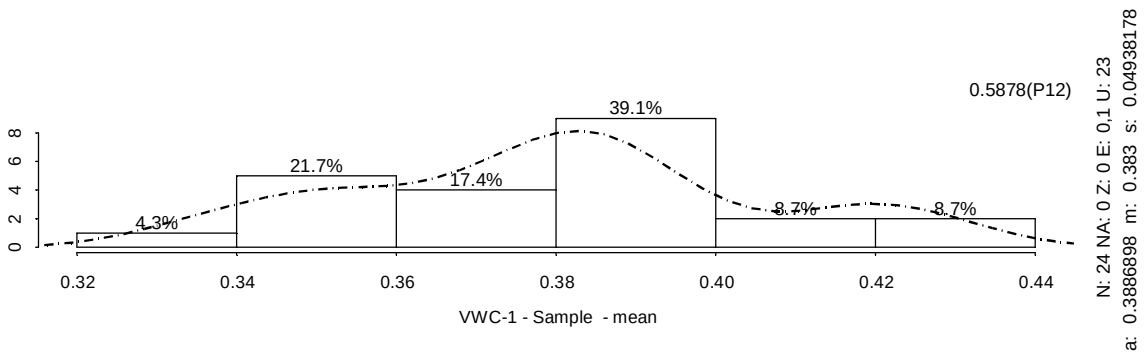

Figure 19: Histogram of the means of the volumetric soil water content at -1 kPa (pF 1.0) (m^3m^{-3})

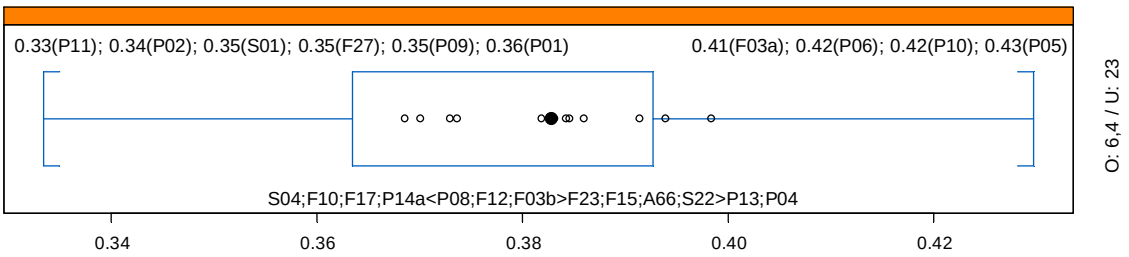

Figure 20: Box plot of the means of the volumetric soil water content at -1 kPa (pF 1.0) (m^3m^{-3})

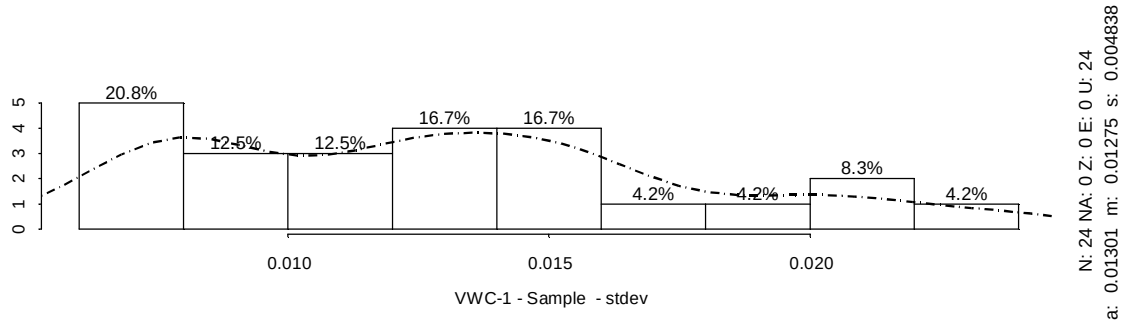

Figure 21: Histogram of the standard deviations of the volumetric soil water content at -1 kPa (pF 1.0) (m^3m^{-3})

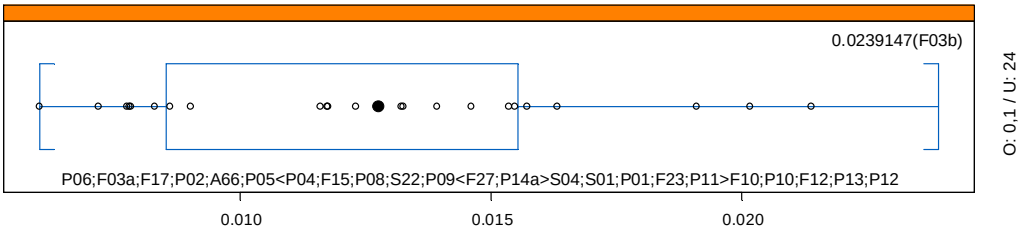

Figure 22: Box plot of the standard deviations of the volumetric soil water content at -1 kPa (pF 1.0) (m^3m^{-3})

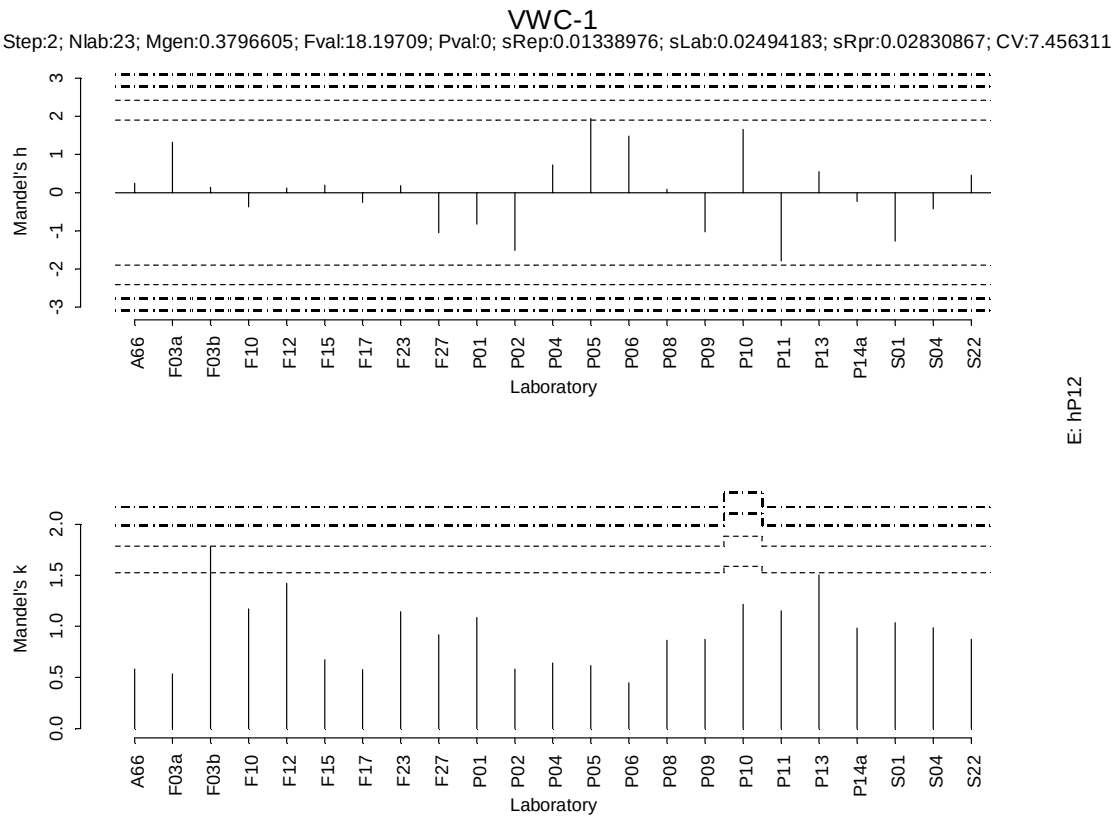

Figure 23: Mandel's h and k plot of the volumetric soil water content at –1 kPa (pF 1.0) (m^3m^{-3})

Volumetric water content at -5 kPa (pF 1.7)

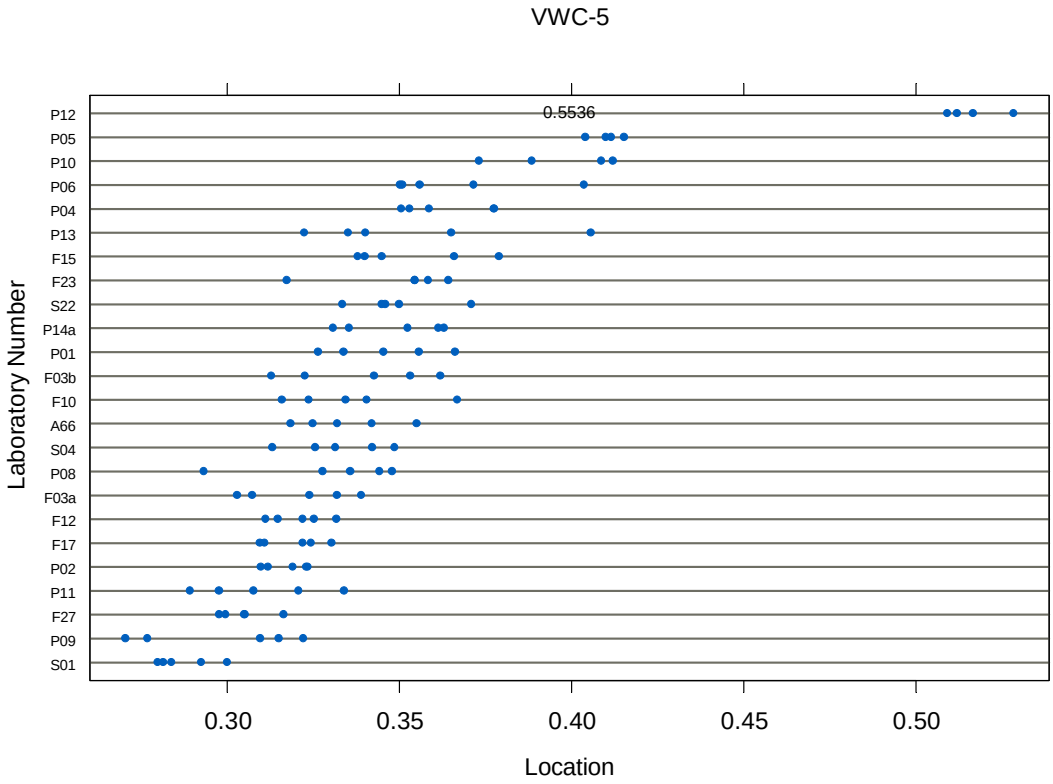

Figure 24: Dot plot of the volumetric soil water content at -5 kPa (pF 1.7) (m^3m^{-3})

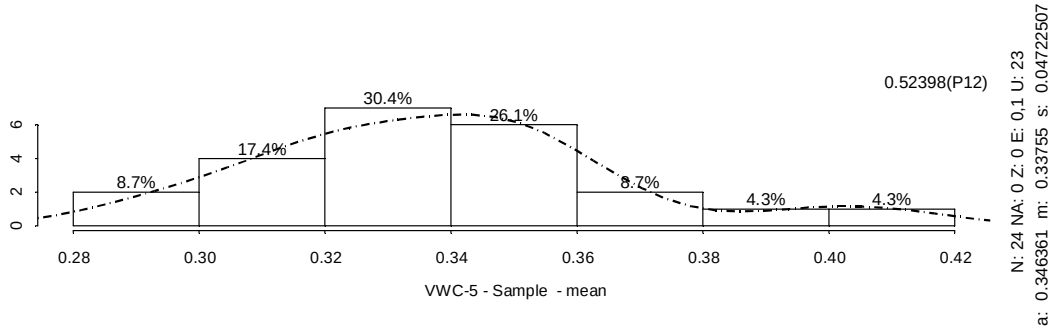

Figure 25: Histogram of the means of the volumetric soil water content at -5 kPa (pF 1.7) (m^3m^{-3})

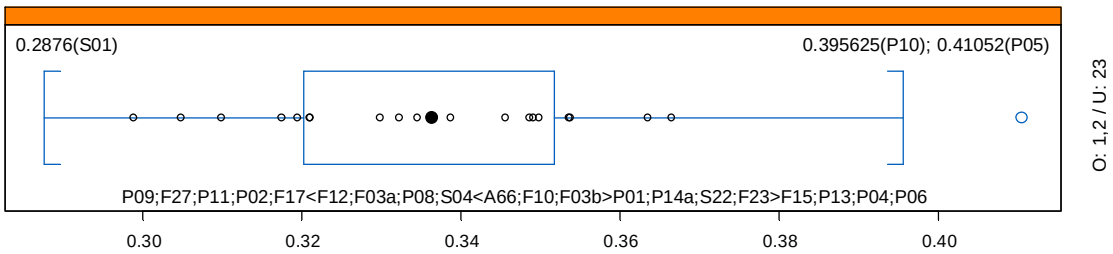

Figure 26: Box plot of the means of the volumetric soil water content at -5 kPa (pF 1.7) (m^3m^{-3})

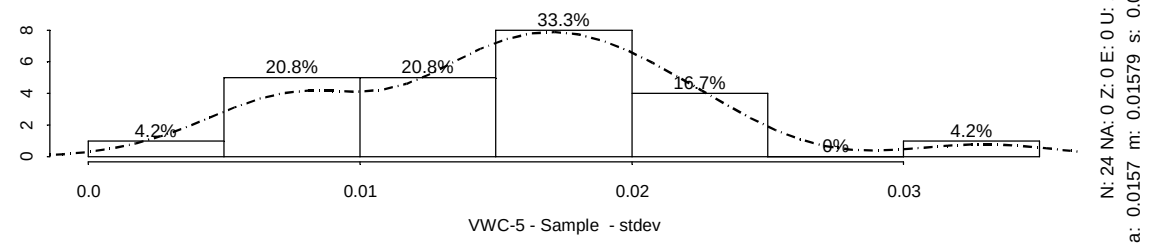

Figure 27: Histogram of the standard deviations of the volumetric soil water content at -5 kPa (pF 1.7) (m^3m^{-3})

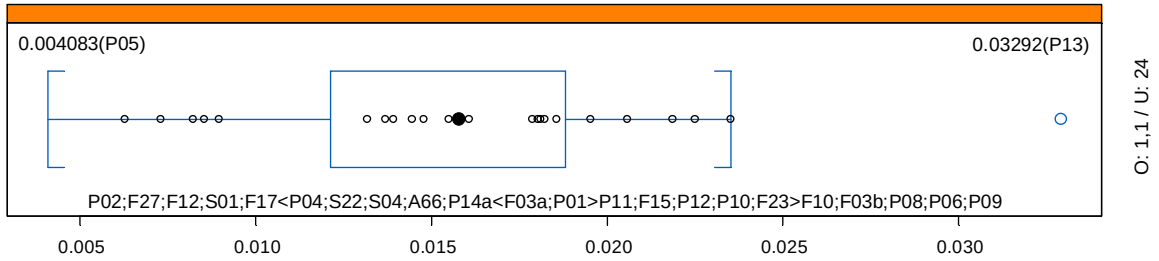

Figure 28: Box plot of the standard deviations of the volumetric soil water content at -5 kPa (pF 1.7) (m^3m^{-3})

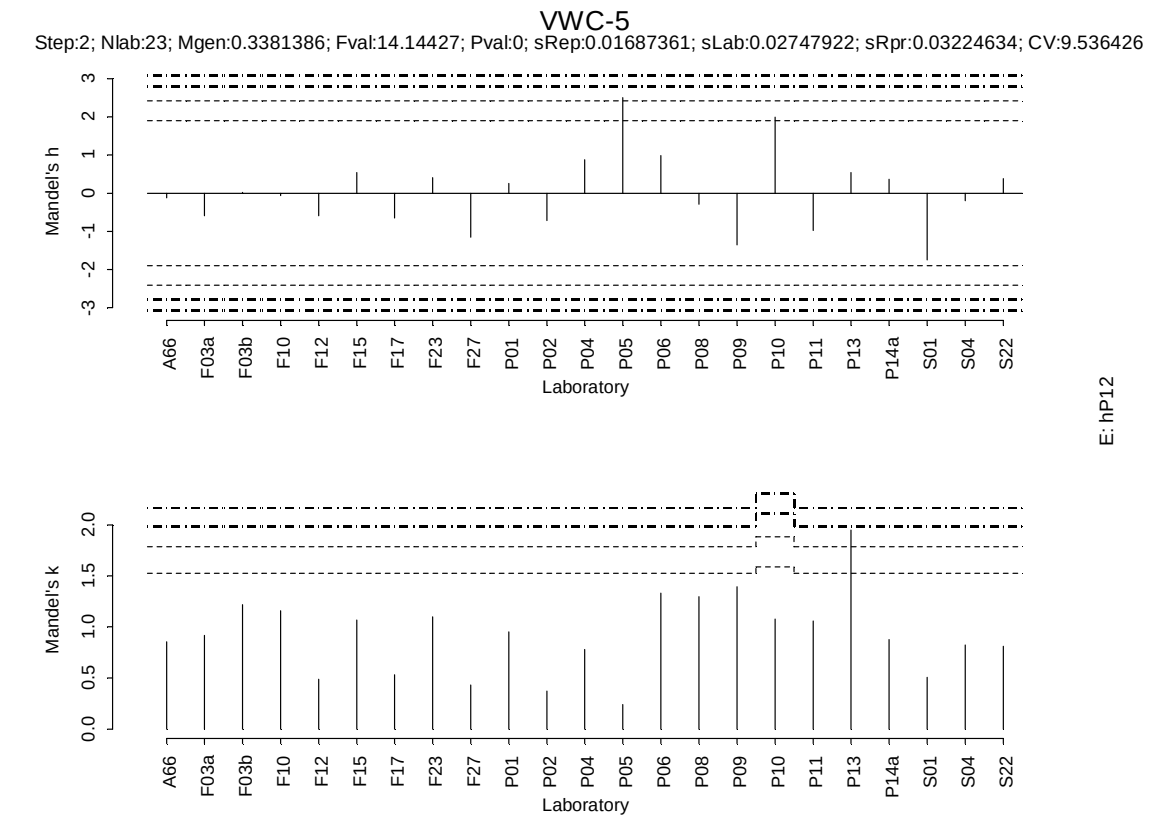

Figure 29: Mandel's h and k plot of the volumetric soil water content at -5 kPa (pF 1.7) (m^3m^{-3})

Volumetric water content at -10 kPa (pF 2.0)

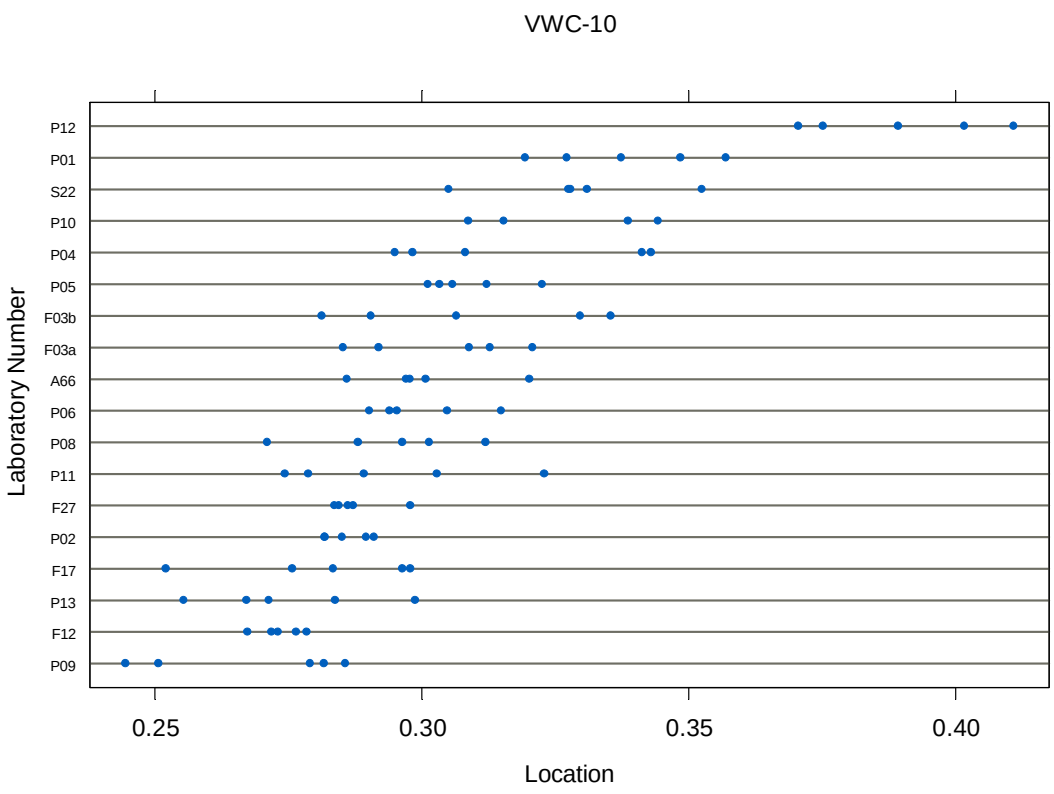

Figure 30: Dot plot of the volumetric soil water content at -10 kPa (pF 2.0) (m^3m^{-3})

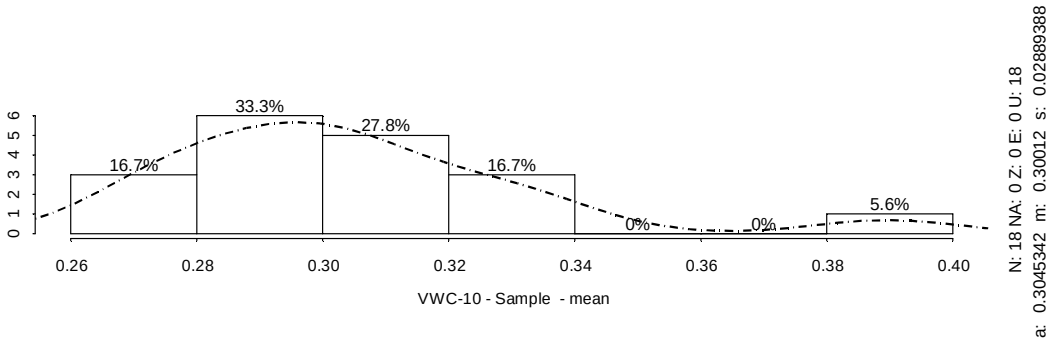

Figure 31: Histogram of the means of the volumetric soil water content at -10 kPa (pF 2.0) ($m^3.m^{-3}$)

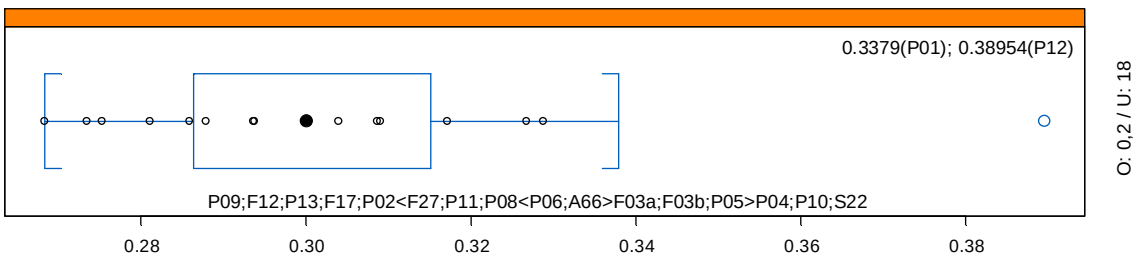

Figure 32: Box plot of the means of the volumetric soil water content at -10 kPa (pF 2.0) (m^3m^{-3})

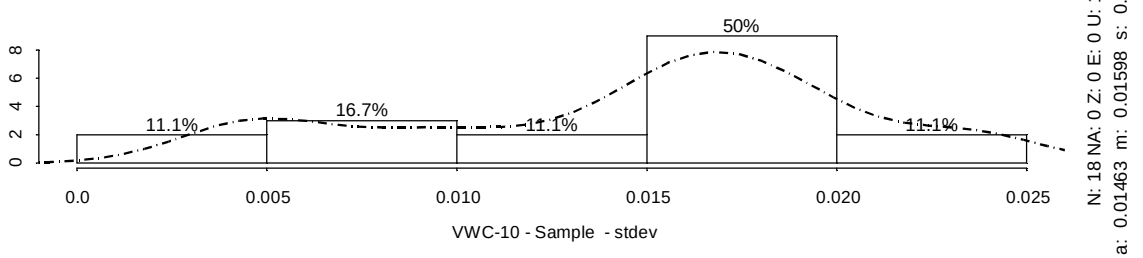

Figure 33: Histogram of the standard deviations of the volumetric soil water content at -10 kPa (pF 2.0) (m^3m^{-3})

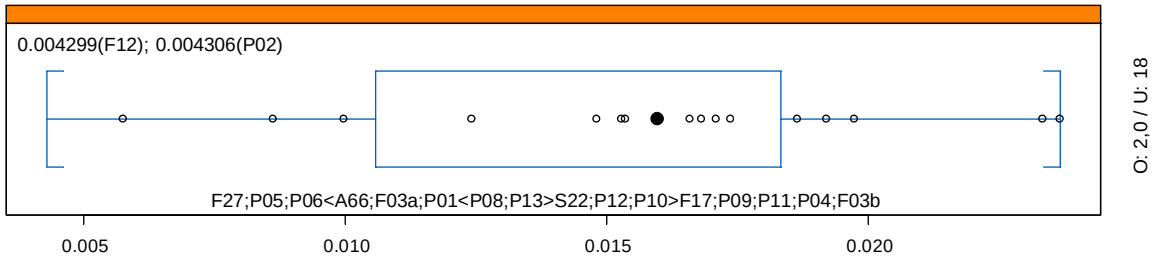

Figure 34: Box plot of the standard deviations of the volumetric soil water content at -10 kPa (pF 2.0) (m^3m^{-3})

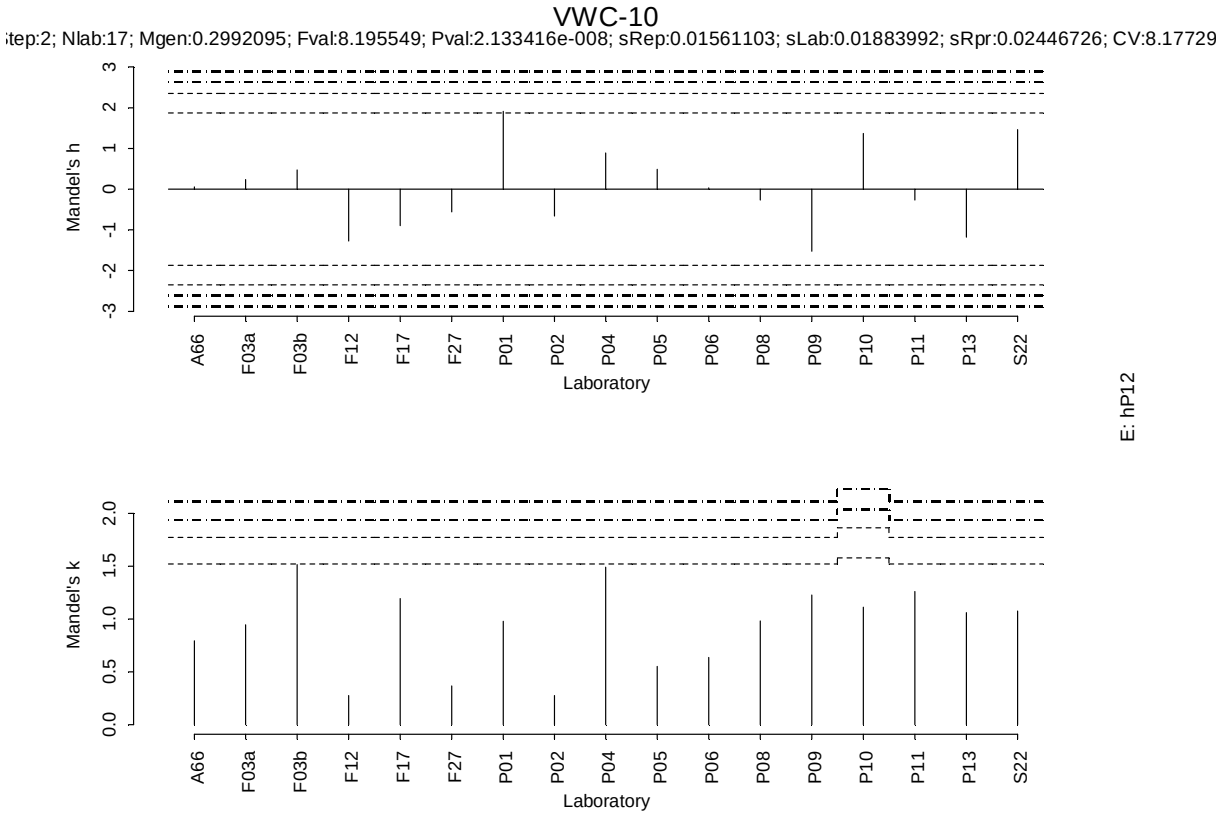

Figure 35: Mandel's h and k plot of the volumetric soil water content at -10 kPa (pF 2.0) (m^3m^{-3})

Volumetric water content at -33 kPa (pF 2.5)

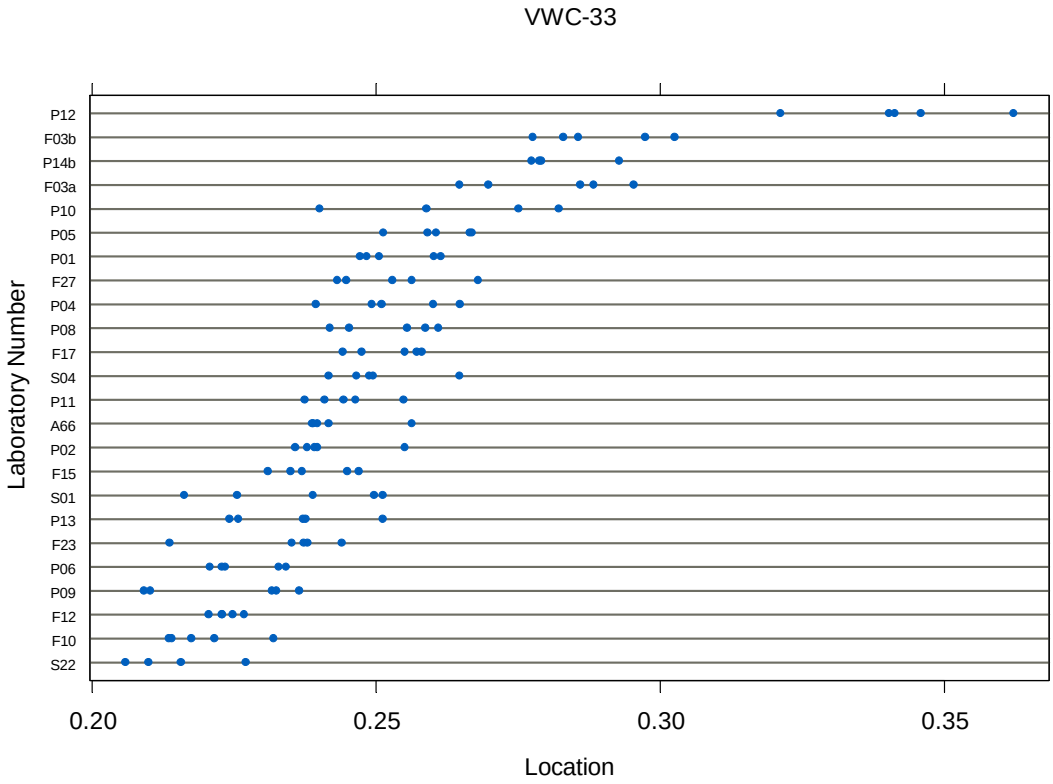

Figure 36: Dot plot of the volumetric soil water content at -33 kPa (pF 2.5) (m^3m^{-3})

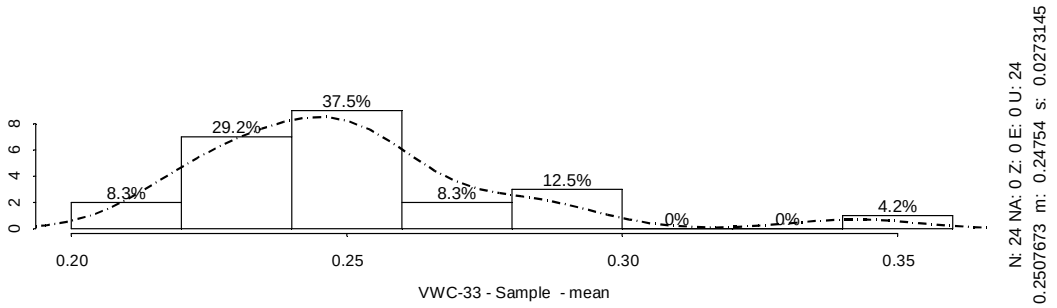

Figure 37: Histogram of the means of the volumetric soil water content at -33 kPa (pF 2.5) (m^3m^{-3})

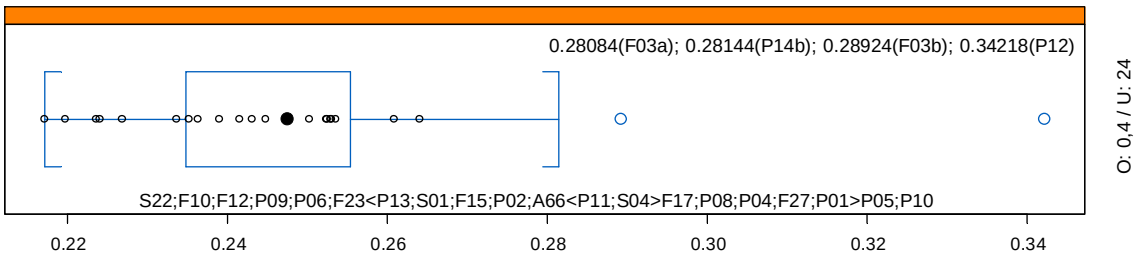

Figure 38: Box plot of the means of the volumetric soil water content at -33 kPa (pF 2.5) (m^3m^{-3})

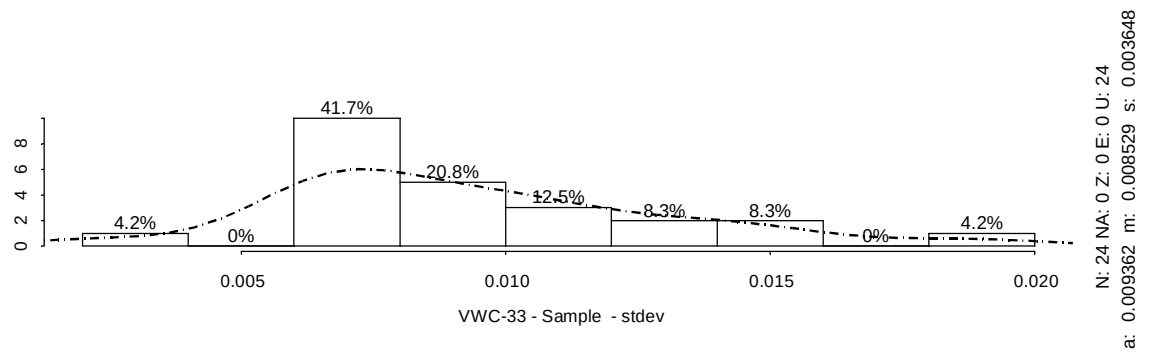

Figure 39: Histogram of the standard deviations of the volumetric soil water content at -33 kPa (pF 2.5) (m^3m^{-3})

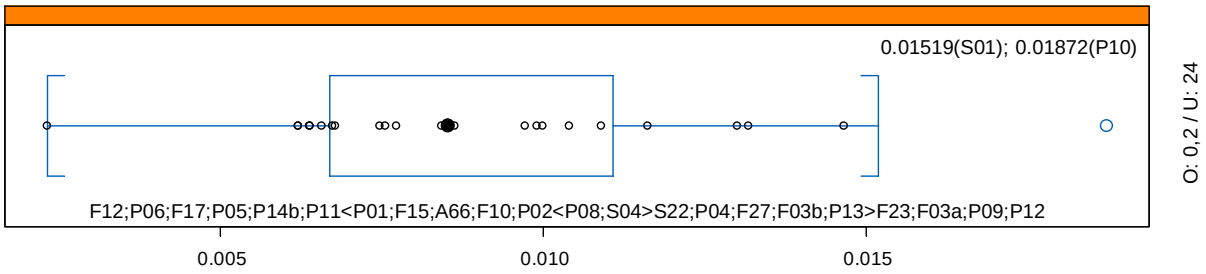

Figure 40: Box plot of the standard deviations of the volumetric soil water content at -33 kPa (pF 2.5) (m^3m^{-3})

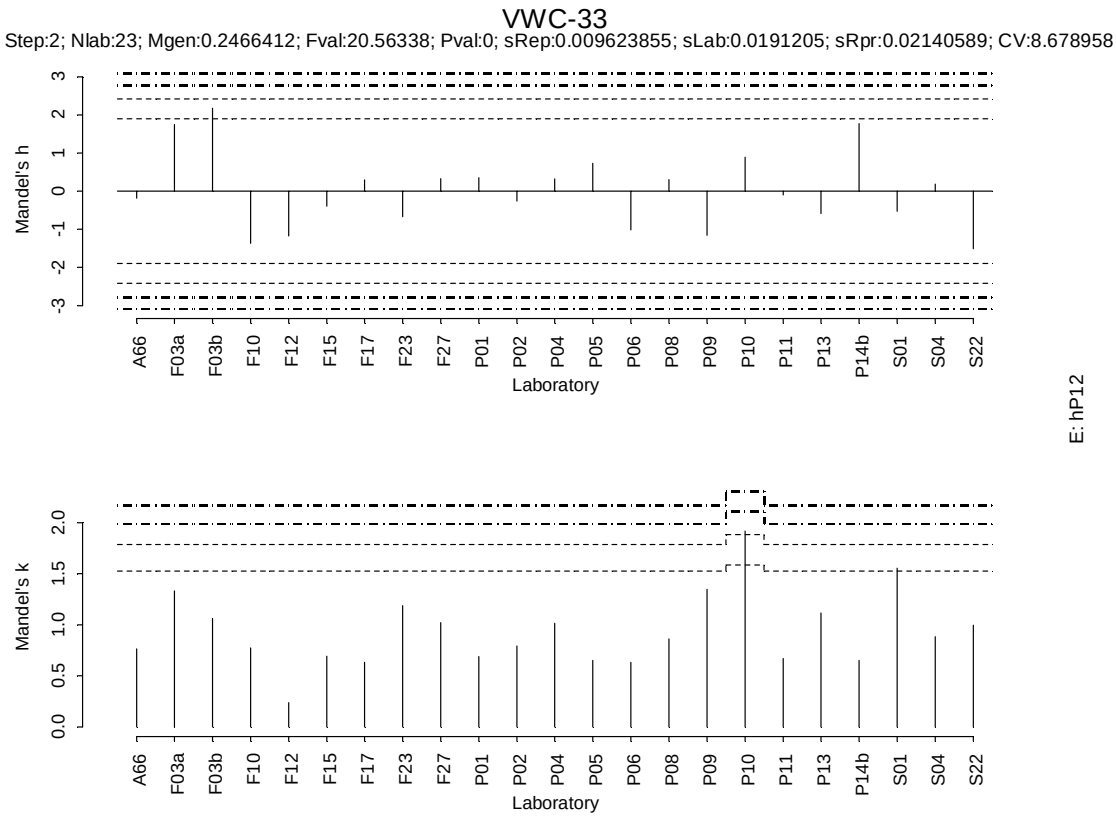

Figure 41: Mandel's h and k plot of the volumetric soil water content at -33 kPa (pF 2.5) (m^3m^{-3})

Volumetric water content at -100 kPa (pF 3.0)

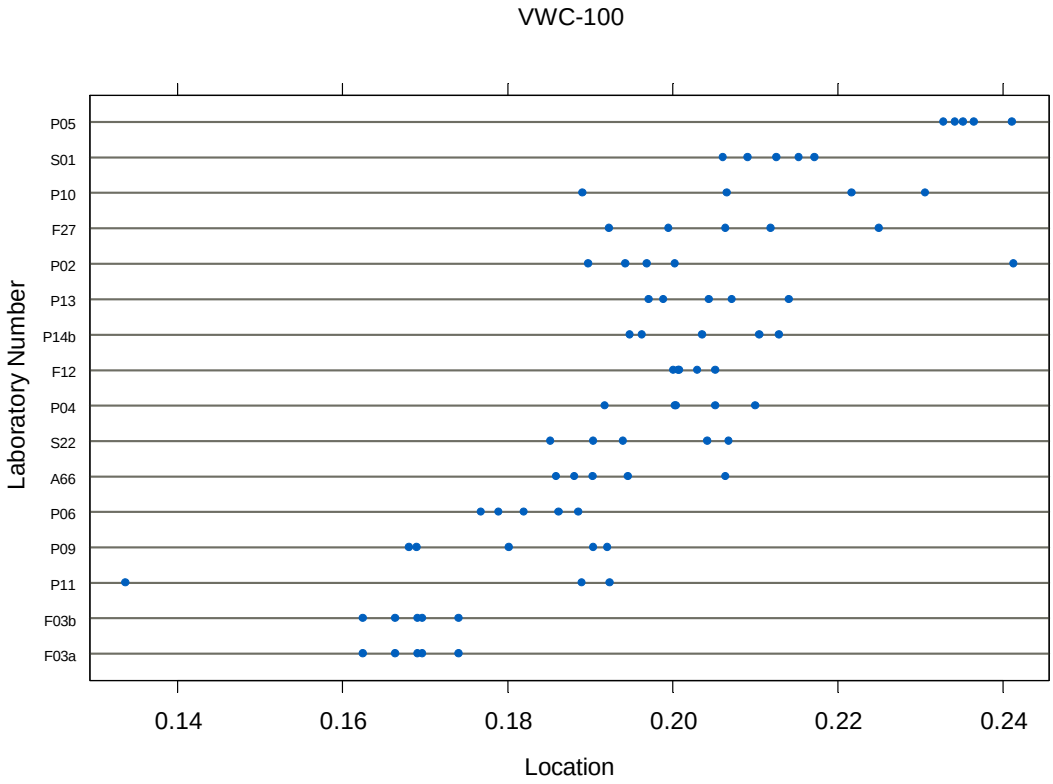

Figure 42: Dot plot of the volumetric soil water content at -100 kPa (pF 3.0) (m^3m^{-3})

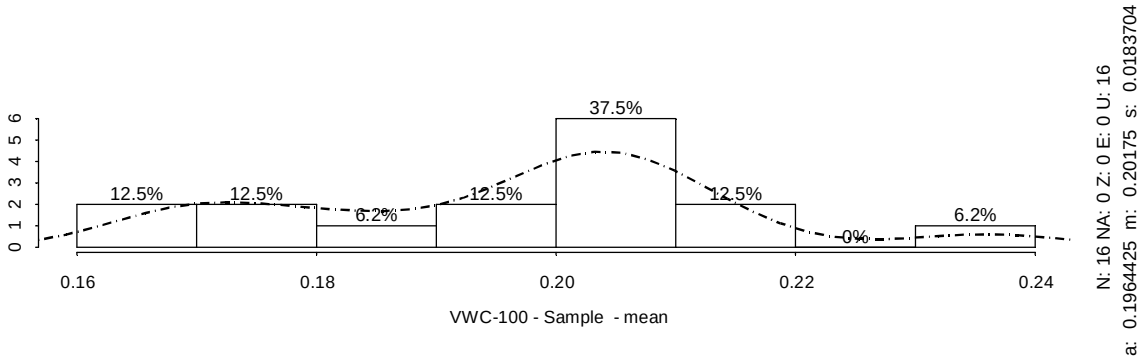

Figure 43: Histogram of the means of the volumetric soil water content at -100 kPa (pF 3.0) (m^3m^{-3})

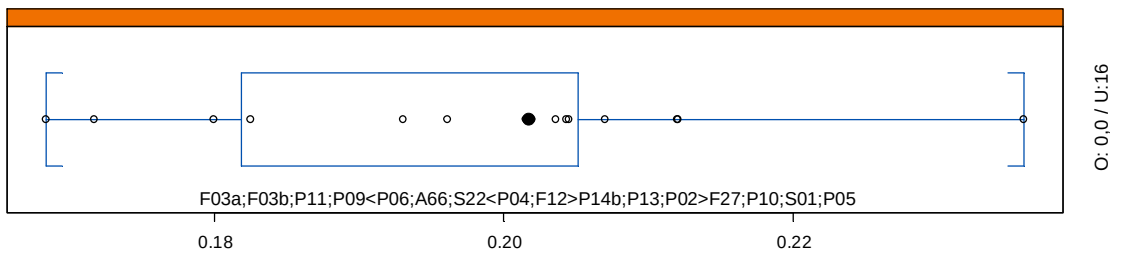

Figure 44: Box plot of the means of the volumetric soil water content at -100 kPa (pF 3.0) (m^3m^{-3})

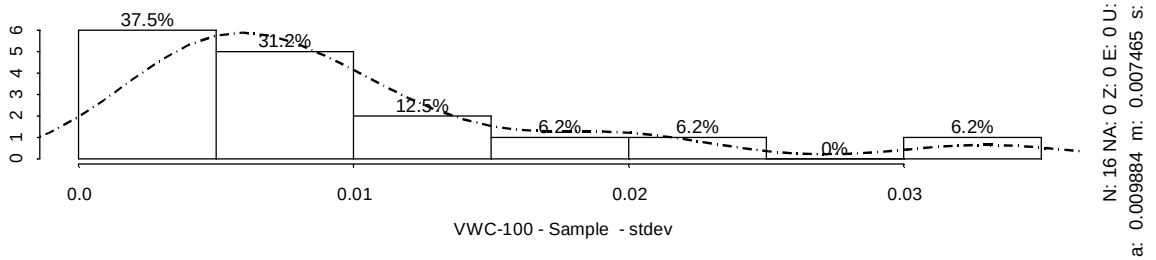

Figure 45: Histogram of the standard deviations of the volumetric soil water content at -100 kPa (pF 3.0) (m^3m^{-3})

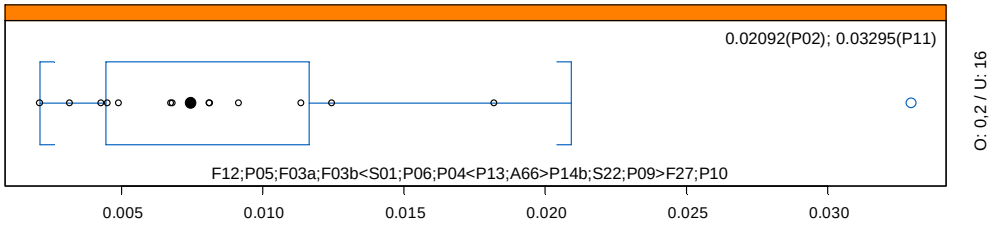

Figure 46: Box plot of the standard deviations of the volumetric soil water content at -100 kPa (pF 3.0) (m^3m^{-3})

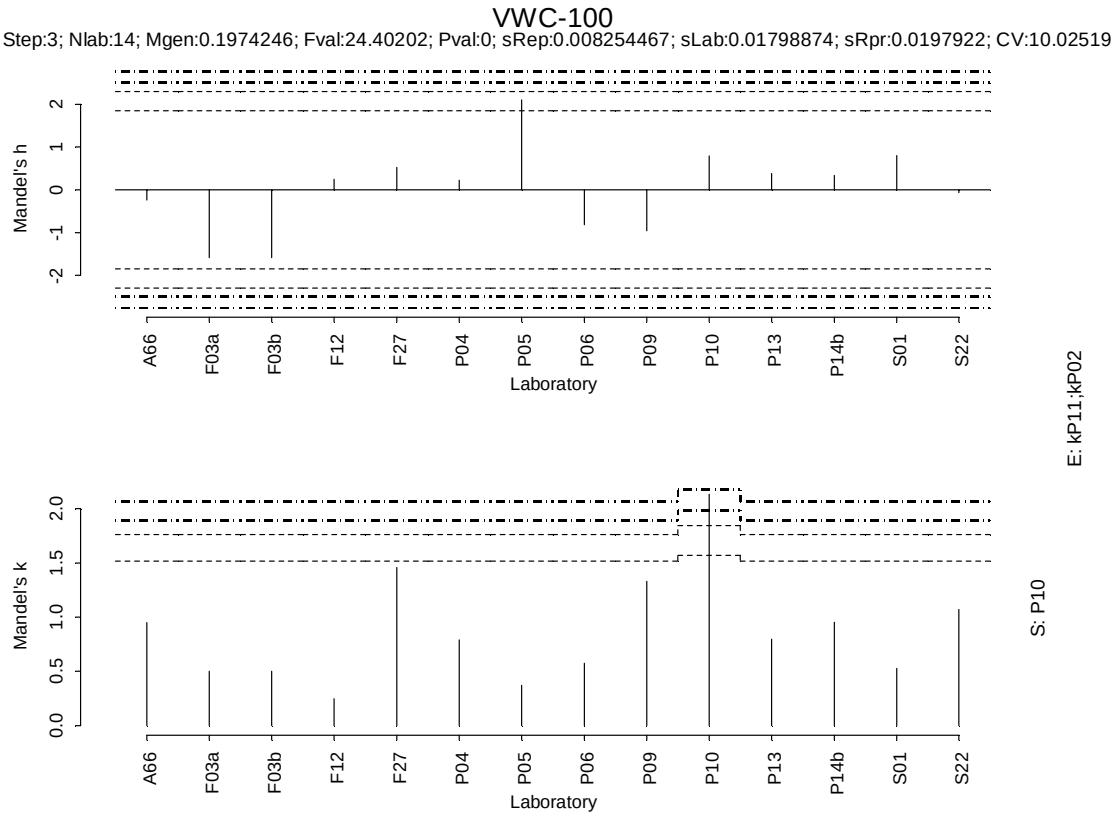

Figure 47: Mandel's h and k plot of the volumetric soil water content at -100 kPa (pF 3.0) (m^3m^{-3})

Volumetric water content at -250 kPa (pF 3.4)

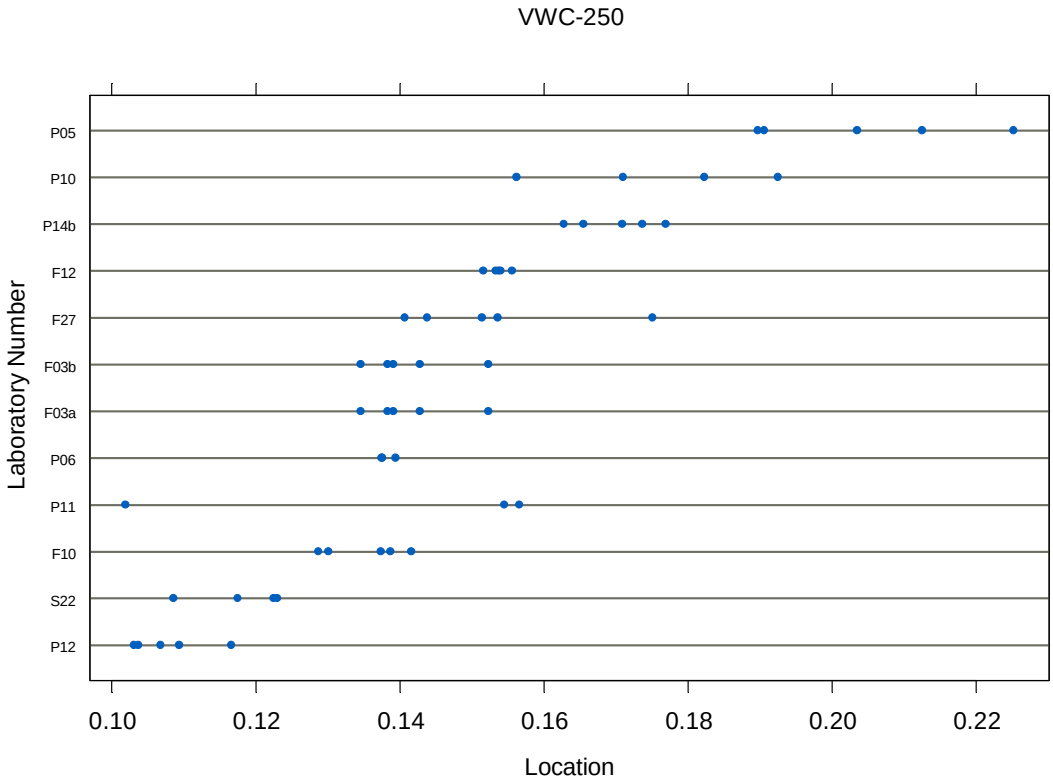

Figure 48: Dot plot of the volumetric soil water content at -250 kPa (pF 3.4) (m^3m^{-3})

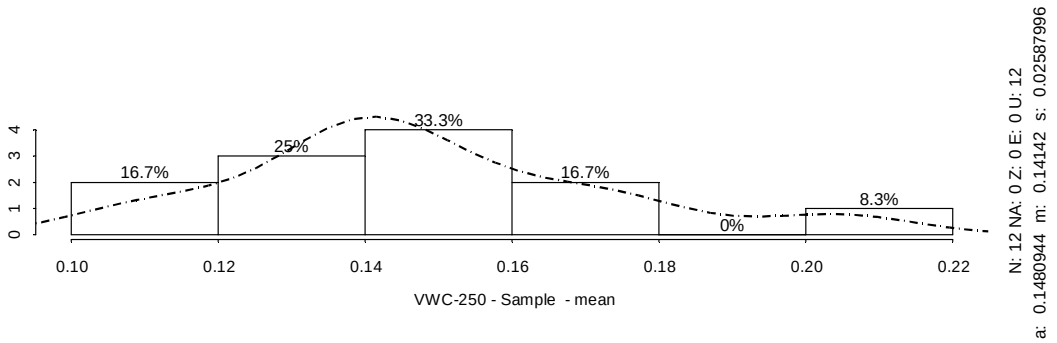

Figure 49: Histogram of the means of the volumetric soil water content at -250 kPa (pF 3.4) (m^3m^{-3})

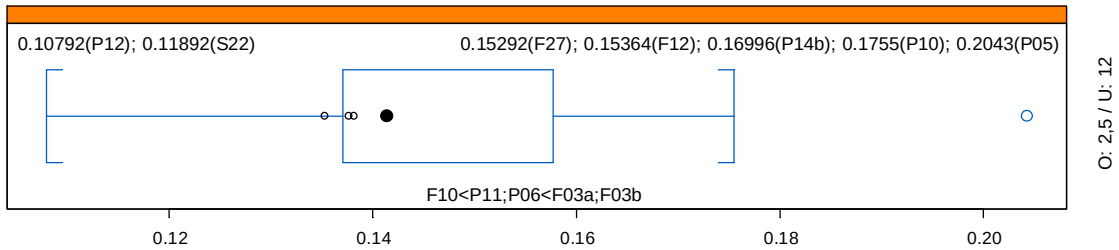

Figure 50: Box plot of the means of the volumetric soil water content at -250 kPa (pF 3.4) (m^3m^{-3})

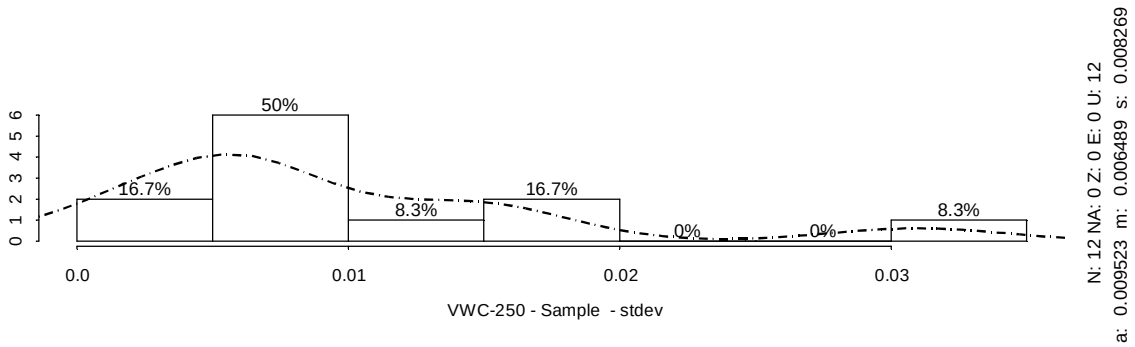

Figure 51: Histogram of the standard deviations of the volumetric soil water content at -250 kPa (pF 3.4) (m^3m^{-3})

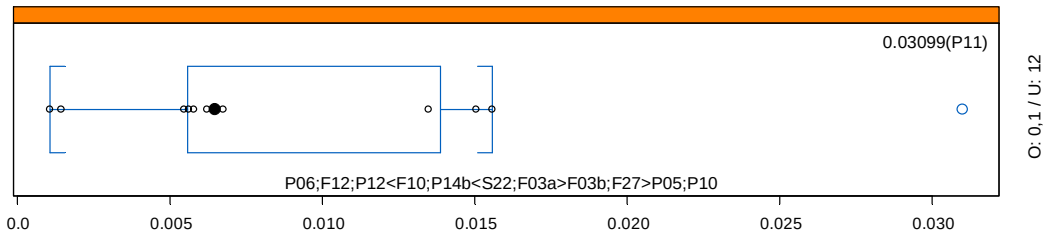

Figure 52: Box plot of the standard deviations of the volumetric soil water content at -250 kPa (pF 3.4) (m^3m^{-3})

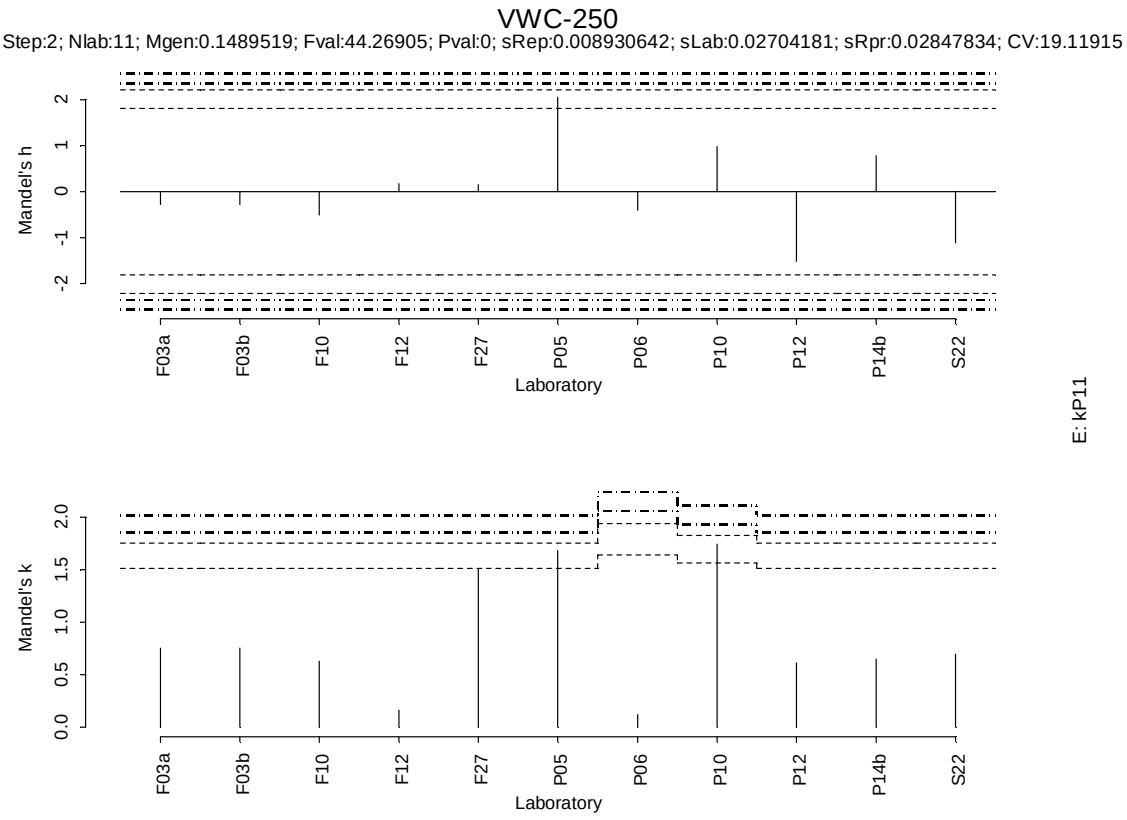

Figure 53: Mandel's h and k plot of the volumetric soil water content at -250 kPa (pF 3.4) (m^3m^{-3})

Volumetric water content at -1500 kPa (pF 4.2)

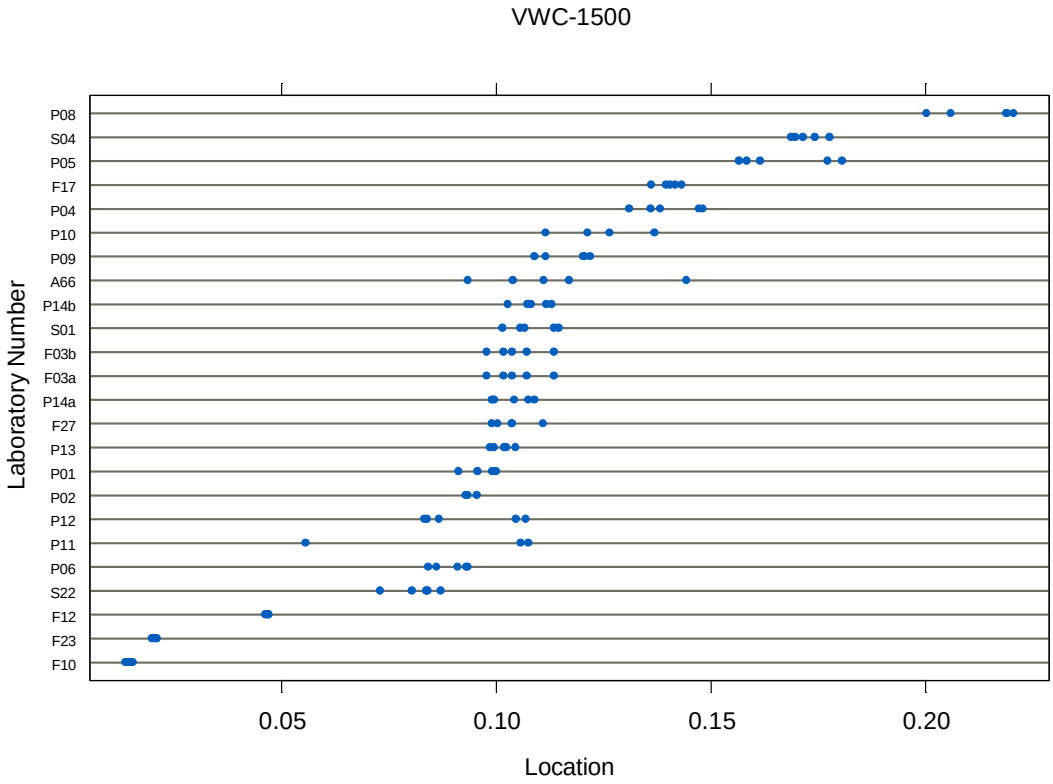

Figure 54: Dot plot of the volumetric soil water content at -1500 kPa (pF 4.2) (m^3m^{-3})

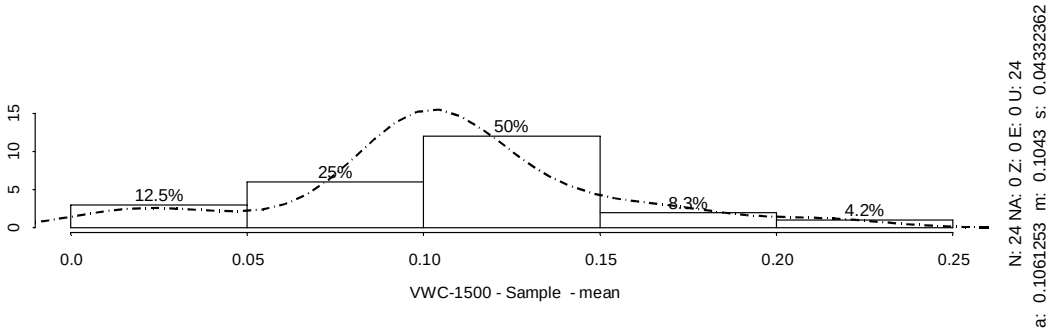

Figure 55: Histogram of the means of the volumetric soil water content at -1500 kPa (pF 4.2) (m^3m^{-3})

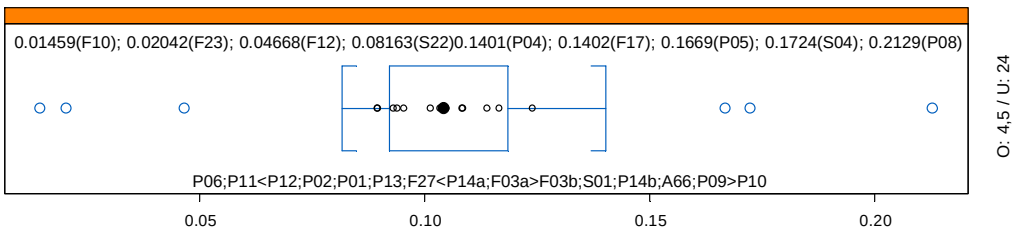

Figure 56: Box plot of the means of the volumetric soil water content at -1500 kPa (pF4.2) (m^3m^{-3})

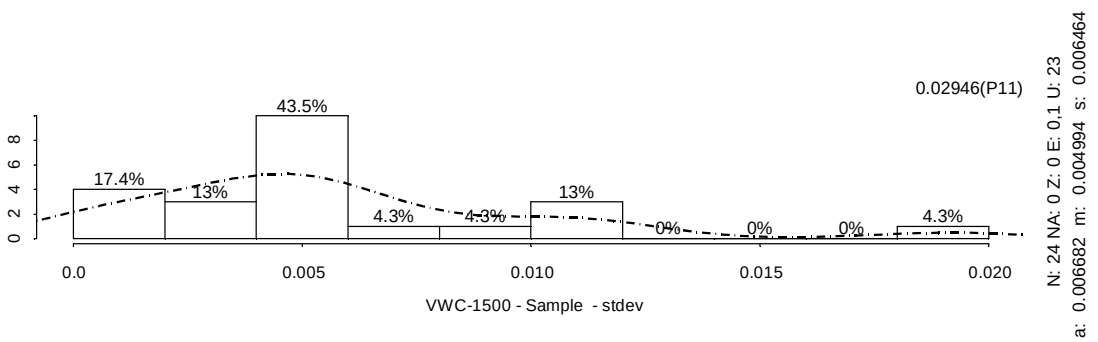

Figure 57: Histogram of the standard deviations of the volumetric soil water content at -1500 kPa (pF 4.2) (m^3m^{-3})

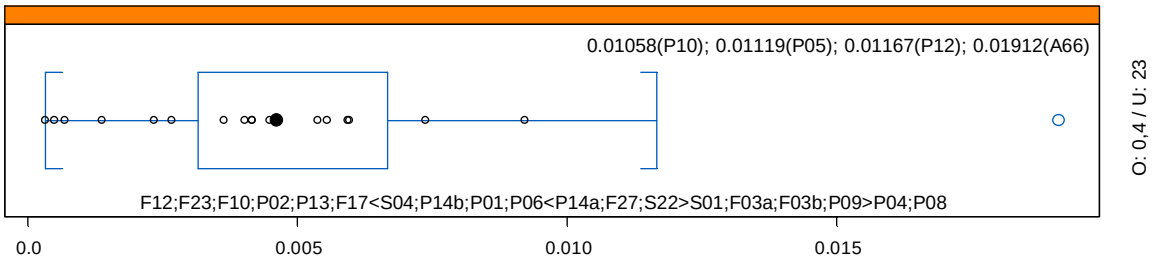

Figure 58: Box plot of the standard deviations of the volumetric soil water content at -1500 kPa (pF 4.2) (m^3m^{-3})

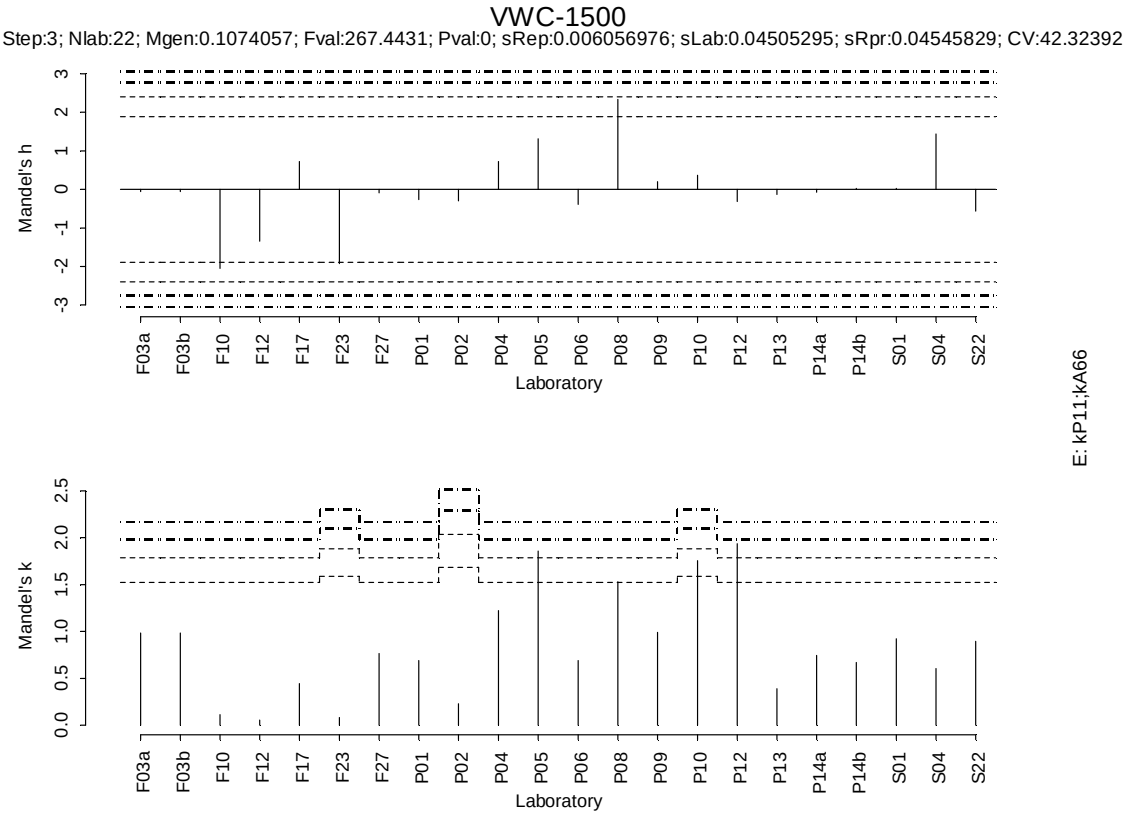

Figure 59: Mandel's h and k plot of the volumetric soil water content at -1500 kPa (pF 4.2) (m^3m^{-3})

Dry soil bulk density (kg.m^{-3})

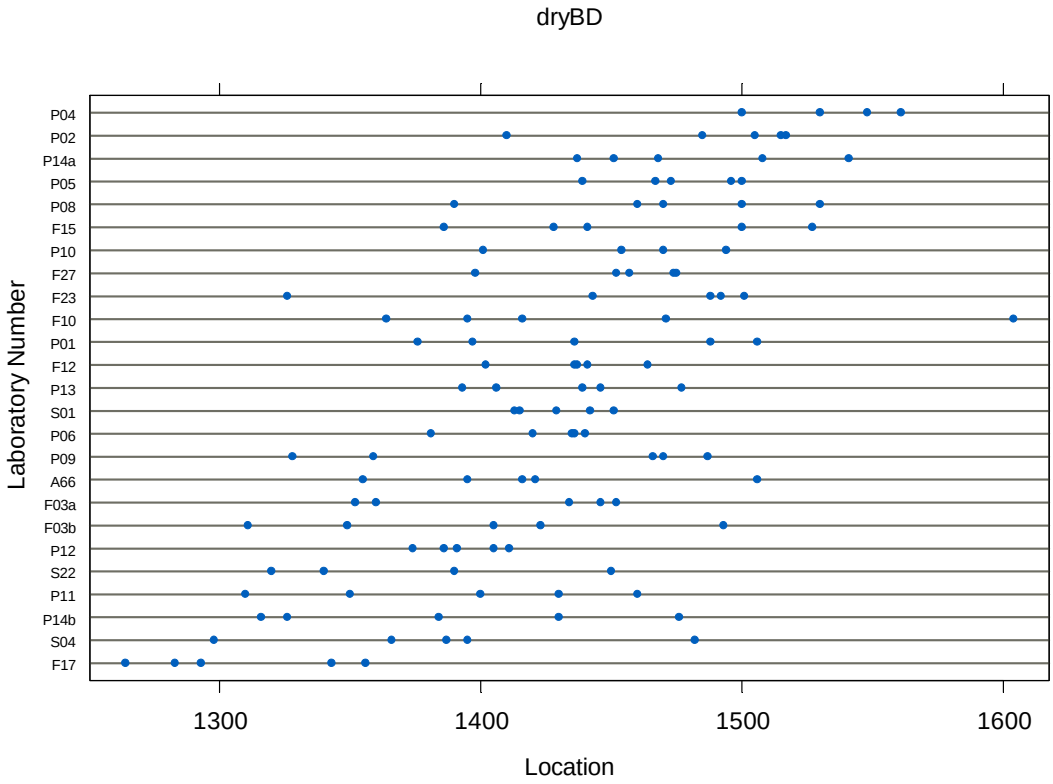

Figure 60: Dot plot of the dry soil bulk density (kg m^{-3})

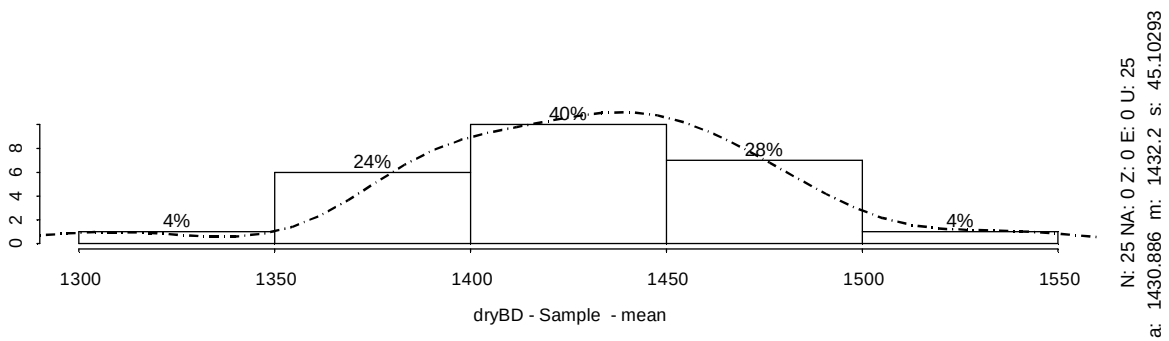

Figure 61: Histogram of the means of the dry soil bulk density (kg m^{-3})

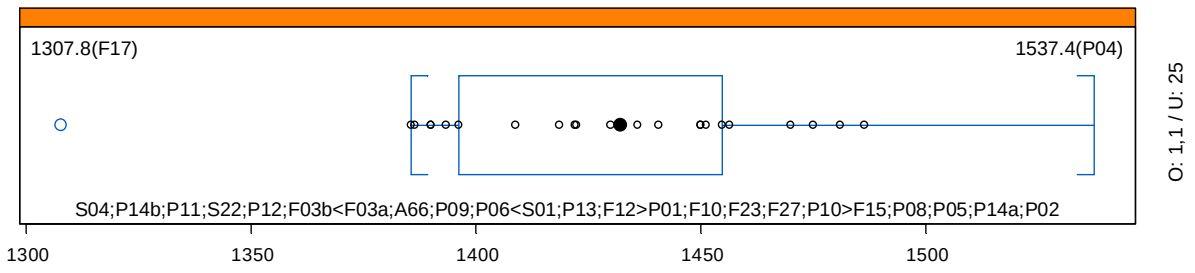

Figure 62: Box plot of the means of dry soil bulk density (kg m^{-3})

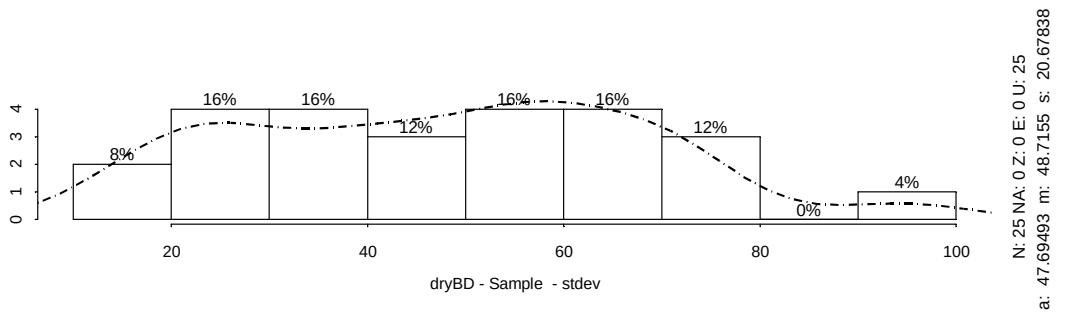

Figure 63: Histogram of the standard deviations of the dry soil bulk density (kg m^{-3})

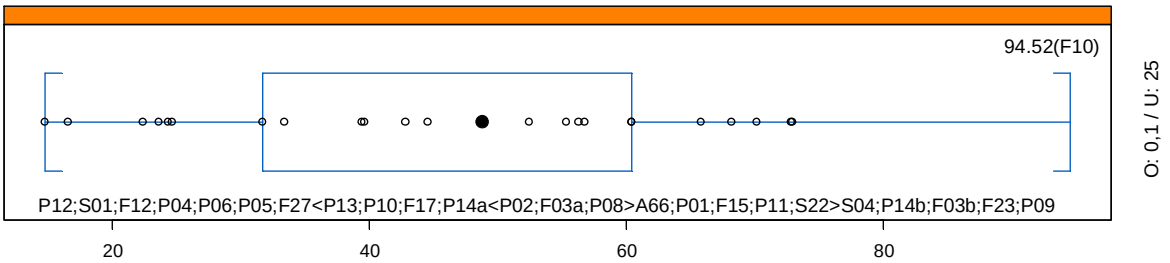

Figure 64: Box plot of the standard deviations of the dry soil bulk density (kg m^{-3})

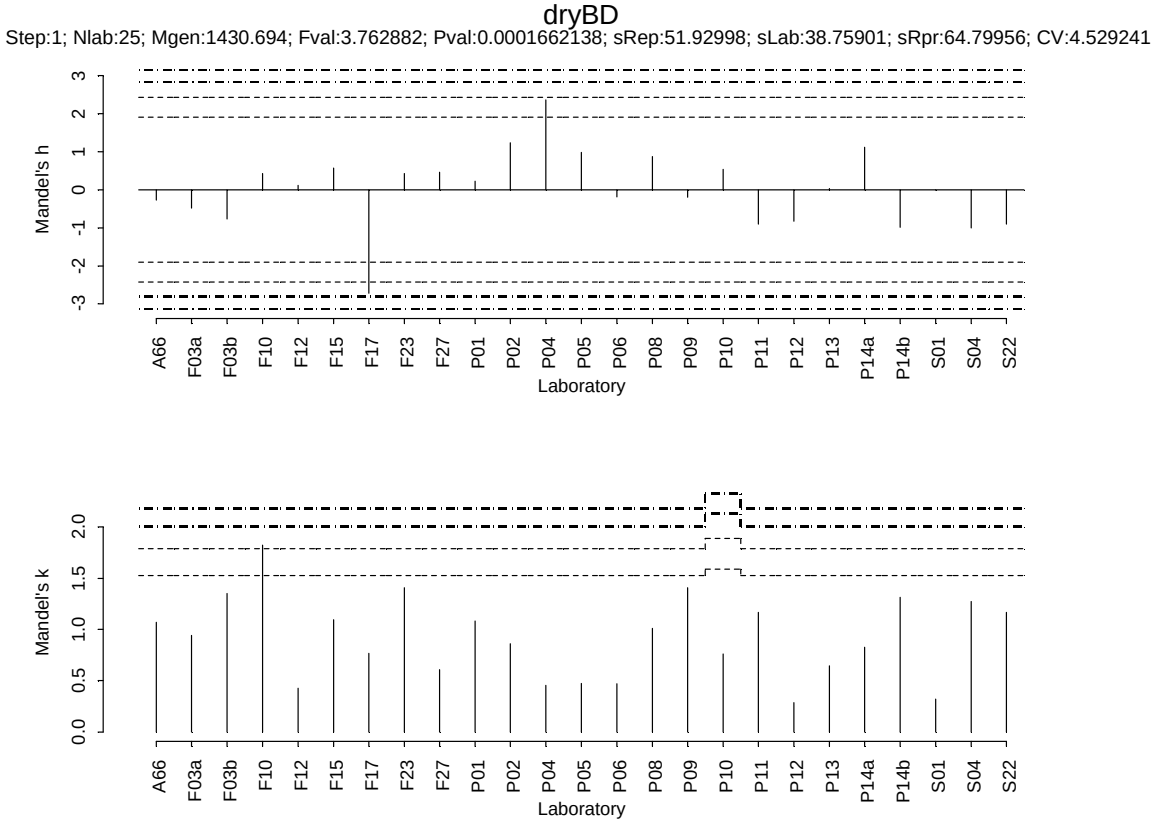

Figure 65: Mandel's h and k plot of the dry soil bulk density (kg m^{-3})