



## Interlaboratory comparison of soil physical parameters

### Authors

Bernhard Buchter, Alberta GmbH, Zürich, Switzerland

Markus Berli, Desert Research Institute, Las Vegas, USA

Peter Weisskopf, Marlies Sommer, Agroscope, Switzerland



## Impressum

Editor:	Agroscope, Institute for Sustainability Sciences ISS Reckenholzstrasse 191, 8046 Zurich, Switzerland <a href="http://www.agroscope.ch">www.agroscope.ch</a>
Information:	Peter Weisskopf e-mail: <a href="mailto:peter.weisskopf@agroscope.admin.ch">peter.weisskopf@agroscope.admin.ch</a>
Cover:	Sampling area of the interlaboratory comparison Marties Sommer, Agroscope
Proofreading:	Regula Wolz
Download:	<a href="http://www.agroscope.ch/science">www.agroscope.ch/science</a>
Copyright:	© Agroscope 2015
ISSN:	2296-729X
ISBN:	978-3-905667-96-7

Project of the Federal Office for the Environment FOEN «Verdichtung – Messmethoden – Normierungsarbeiten», Contract No. 07.0125.PJ / H202-0480

We wish to thank the colleagues from the participating laboratories for their technical contributions and the constructive cooperation, and the project companions J.-P. Clement (as representative of the Federal Office for the Environment FOEN), S. Tobias (as representative of the executive board of the Swiss Soil Science Society SSSS) as well as D. Schaub and W.G. Sturny (as representatives of the Soil Protection Offices of the Cantons) for their support.

# Table of contents

<b>Summary</b> .....	<b>4</b>
<b>1 Introduction</b> .....	<b>5</b>
<b>2 Materials and methods</b> .....	<b>6</b>
2.1 Laboratories and soil .....	6
2.2 Procedures .....	6
2.3 Methods and parameters.....	7
2.3.1 Extraction of soil samples .....	7
2.3.2 Preparation of soil samples .....	7
2.3.3 Analysis of soil samples .....	7
2.4 Statistical analysis .....	8
<b>3 Results and discussion</b> .....	<b>9</b>
3.1 Bulk density .....	9
3.2 Macropore volume .....	10
3.3 Total pore volume.....	14
3.4 Particle density .....	15
3.5 Saturated hydraulic conductivity .....	17
<b>4 Summary</b> .....	<b>18</b>
4.1 Bulk density .....	18
4.2 Macropore volume .....	18
4.3 Total pore volume.....	19
4.4 Saturated hydraulic conductivity .....	19
<b>5 Conclusions</b> .....	<b>19</b>
<b>References</b> .....	<b>20</b>
<b>List of figures</b> .....	<b>21</b>

## Summary

**This report summarizes the first interlaboratory comparison of soil physical parameters carried out in Switzerland. The overall aims were (i) to quantify the influence of soil sample extraction, preparation and analysis on measured values of soil physical parameters; and (ii) to assess the accuracy of standardized methods, with the ultimate objective of characterizing measured values of soil physical parameters together with estimates of uncertainty.**

The increased use of heavy equipment for agricultural soil management and during recultivation or construction on agricultural land may lead to harmful compaction of agricultural soils, reducing their fertility for years or even decades if the subsoil is compacted.

With the target and intervention values proposed by the Swiss Soil Science Society (Document 13, 2004), quantitative guidelines to assess the state of soil structure using soil physical parameters such as bulk density, macropore volume, saturated hydraulic conductivity and penetration resistance are now in place and provide a legal basis for the protection of soils against mechanical impacts.

The methods used in the past to analyze soil physical parameters were mainly developed by research laboratories to solve specific research questions; the results obtained with these methods did not need to be comparable between laboratories.

This report summarizes the first interlaboratory comparison of soil physical parameters carried out in Switzerland, involving 10 laboratories offering soil physical analyses.

On a site near Möriswil (Canton Bern, Switzerland), the topsoil of a Cambisol (WRB, 2006) under grass was sampled according to four different procedures. These procedures allowed for separating the effects of sampling, sample preparation and sample analysis by the laboratories and at the same time for comparing the results between the laboratories.

The methods of the participating laboratories to sample, prepare and analyze soil samples for bulk density, total porosity, macroporosity and saturated hydraulic conductivity are described and compared. The results of the laboratories considering the different procedures are presented and discussed.

This interlaboratory comparison led to the conclusions that

- (i) the values obtained for bulk density, macropore volume, total pore volume and saturated hydraulic conductivity in soil samples were significantly different when more than one laboratory was involved;
- (ii) the influence of the analysis phase in the laboratory was more pronounced than that of extracting and preparing samples, and much greater than that of soil heterogeneity in the sampling areas;
- (iii) measured values have to be accompanied by values characterizing the uncertainty caused by the measurement process, necessitating a thorough error propagation analysis of the methods and procedures used;
- (iv) the analytical methods for determining these soil physical parameters have to be improved and standardized, particularly that for measuring saturated hydraulic conductivity;
- (v) there is an urgent need for reference samples to check laboratory procedures thoroughly, allowing reproducible measurements of porosity and conductivity for interlaboratory comparisons.

# 1 Introduction

Harmful soil compaction is a growing concern in agriculture. Heavy wheel loads can damage the soil structure by altering porosity, destroying macropores and reducing pore conductivity, leading to reduced oxygen supply for plant roots and soil organisms. Root growth is impaired and hence soil fertility is diminished. Agricultural productivity has been increased in recent decades through the mechanization of cultivation techniques such as soil tillage, crop fertilization and harvesting. However, this mechanization has also increased the likelihood of soil compaction. In addition, during recultivation or construction on agricultural land, other heavy equipment is used, which may lead to harmful compaction of agricultural soils, reducing their fertility for years or even decades if the subsoil is compacted. Likewise in forestry, larger and therefore heavier harvesting equipment is destroying the structure of forest soils.

Until recently, the decision on whether soil fertility was affected or not was left to the personal judgment of soil experts. However, with the target and intervention values proposed by the Swiss Soil Science Society (Document 13, 2004), quantitative guidelines to assess the state of soil structure using soil physical parameters such as bulk density, macropore volume, saturated hydraulic conductivity and penetration resistance are now in place. These provide a legal basis for the protection of soils against mechanical impacts.

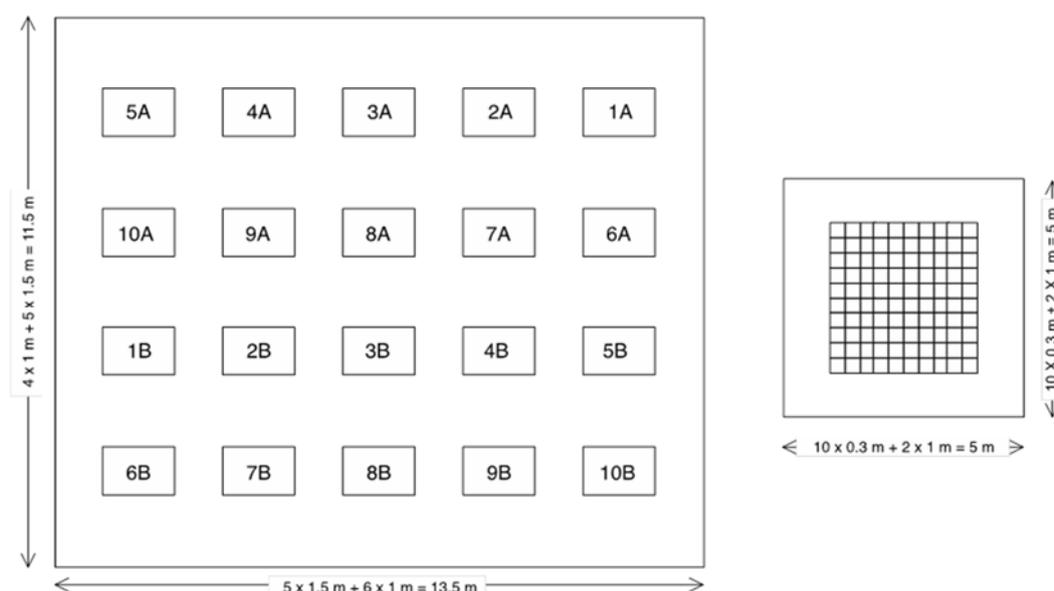
The methods used in the past to analyze soil physical parameters were mainly developed by research laboratories to solve specific research questions. The obtained results did not need to be comparable between laboratories, and analytical costs were seldom limiting. In contrast, the laboratories of public authorities aim to: (i) detect changes in soil physical parameters due to different agricultural management practices; and (ii) assess the soil quality of recultivated land. In these cases, the analytical costs and the comparability of results obtained in different laboratories are usually important criteria. Many private laboratories are also interested in standardized, inexpensive and time-invariant methods.

This report summarizes the first interlaboratory comparison of soil physical parameters carried out in Switzerland. The overall aims were to: (i) quantify the influence of soil sample extraction, preparation and analysis on measured values of soil physical parameters; and (ii) assess the accuracy of standardized methods, with the ultimate objective of characterizing measured values of soil physical parameters together with estimates of uncertainty.

## 2 Materials and methods

### 2.1 Laboratories and soil

Ten laboratories (numbered 1–10) offering soil physical analyses participated in the interlaboratory comparison. One of these, no. 7, was chosen as the reference laboratory. Soil samples were taken from the topsoil of a Cambisol (WRB, 2006) under grass (3rd year ley) near Möriswil (Canton Bern, Switzerland; Swiss coordinates 593.555/203.705, altitude 630 m a.s.l.). The well-aggregated topsoil horizon was free of stones and contained 15% clay and 30–40% silt. Each laboratory, the reference laboratory included, took samples for procedure 1 from sampling area A and for procedure 2 from sampling area B (Figure 1). The reference laboratory took samples for procedures 3 and 4 from the adjacent sampling area C (Figure 1).



**Figure 1** | Left: sampling areas A and B for procedures 1 and 2 by laboratories 1–10. Right: sampling area C for procedures 3 and 4 by reference laboratory 7.

### 2.2 Procedures

The four procedures were as follows:

**Procedure 1:** Comparing laboratories: Each laboratory (L), the reference laboratory (R) included, extracted, prepared and analyzed samples using its own standard methods. The results were given the code LLL or RRR (Table 1).

**Procedure 2:** Assessing the effect of sampling: Each laboratory extracted samples using its own standard methods, but the samples were prepared and analyzed by the reference laboratory. The results were given the code LRR.

**Procedure 3:** Assessing the effect of sample preparation: The reference laboratory extracted the soil samples, and then each laboratory prepared them using its own standard methods and returned them to the reference laboratory, which analyzed them. The results were given the code RLR.

**Procedure 4:** Assessing the effect of sample analysis: The reference laboratory extracted and prepared the samples, and each laboratory analyzed them using its own standard methods. The results were given the code RRL.

**Table 1** | Procedures 1 to 4 for assessing the influence of extracting, preparing and analyzing soil samples. L = work phase done by individual laboratory, R = work phase done by the reference laboratory.

Procedure	Extraction	Preparation	Analysis	Code	Objective
1	L	L	L	LLL	comparing the laboratories
1	R	R	R	RRR	comparing the laboratories
2	L	R	R	LRR	effect of sampling
3	R	L	R	RLR	effect of sample preparation
4	R	R	L	RRL	effect of sample analysis

## 2.3 Methods and parameters

### 2.3.1 Extraction of soil samples

The 10 laboratories used six different sample sizes, namely (diameter × height in mm) 55 × 39, 55 × 42, 80 × 50, 60 × 40, 105 × 110 and 50 × 50. The center of the soil samples was always at a soil depth of 13 cm, irrespective of the sample length. All laboratories used standardized, more or less detailed protocols to take notes on the sampling conditions; five laboratories made an additional pedological assessment of the sampled field soil. All laboratories determined sampling depth from the soil surface using a meter stick. Six laboratories used sample cylinders without a cutting edge. Nine laboratories drove the cylinders into the soil by hammering, one by pressing. The main criteria leading to the rejection of samples were large cracks or holes, large stones, large pores, and incomplete filling of the sample cylinder. All 10 laboratories protected the sample cylinders during subsequent transport and storage by covering the cylinders with lids.

### 2.3.2 Preparation of soil samples

Eight laboratories stored the samples in the refrigerator, in part at controlled humidity, one laboratory used a room with 20 °C and 100% humidity for sample storage and one stored them at room temperature in the basement without controlling temperature and humidity. For preparing the sample surfaces, knives or saws or both were used, with quite different and very individual preparation techniques.

### 2.3.3 Analysis of soil samples

#### *Bulk density*

All laboratories dried the samples at 105–110 °C and determined bulk density ( $D_a$ ) as the ratio of mass of dried soil to sample volume. Sample volume was taken by seven laboratories to be equal to the inner volume of the cylinders and by three laboratories to be the maximum sample volume after water saturation and swelling. Seven laboratories checked bulk density values by comparing them with measured or estimated values of particle density ( $D_r$ ) and total pore volume. Five laboratories recorded the data electronically, five manually on paper.

#### *Total and macropore volumes*

Nine laboratories determined the macropore volume ( $VP_g$ ) at 60 hPa, five of them with commercially available sandboxes and four of them with commercially available pressure cells. Eight laboratories determined the water content at saturation by weighing; six of these assumed that the water content at saturation corresponded to the total pore volume ( $VP_t$ ), whereas the other two calculated total pore volume based on bulk density and particle density. One laboratory determined total pore volume based on measurements of solid/liquid soil substance using an air pycnometer. Macropore volume was then calculated as the difference between water content or total pore volume at water saturation and water content at a matric potential of 60 hPa. The results were checked using different criteria and techniques such as plausibility of data, use of standard samples, monitoring the matric potential of the extraction apparatus, or comparisons with different methodological approaches such as air pycnometer and pressure cell. Two laboratories recorded the data electronically, seven manually on paper.

#### *Particle density*

Particle density was not included as an official parameter in this interlaboratory comparison. However, if data were delivered by the laboratory, they were included in the comparison.

### *Saturated hydraulic conductivity*

In contrast to the other soil physical parameters, the laboratories took samples for determining saturated hydraulic conductivity ( $K_{sat}$ ) according to procedure 1 only; procedures 2, 3 and 4 were not possible due to the incompatibility of the measuring devices and sample sizes.

Seven laboratories determined saturated hydraulic conductivity. Three used a commercially available permeameter with a sample height of 39 mm, two used custom-made permeameters with a sample height of 42 mm, and one used a custom-made permeameter with a sample height of 100 mm. The latter applied the falling head method, whereas the other five laboratories used the constant head method. The remaining laboratory used a triaxial-shear testing machine.

All laboratories checked their results by repeating the measurements, some by verifying the pressure head, but only one by comparing the measured values against the minimum and maximum hydraulic conductivity of the permeameter and calculating the uncertainty of the results. Data were recorded manually on paper.

The laboratories used the following units for hydraulic conductivity: cm/s, m/s, cm/d and m/d. The results of this interlaboratory comparison were all converted to, and are reported as,  $pK_{sat}$  values ( $= -\log_{10}(K_{sat})$ ).

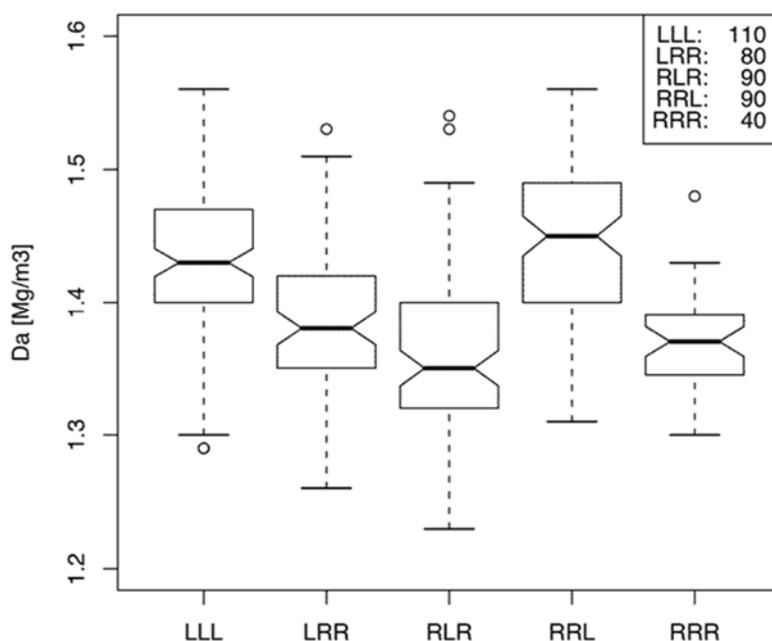
## 2.4 Statistical analysis

Statistical analysis and plotting of results were performed using the software R (R Development Core Team, 2008). The results are shown using box plots. The length of the boxes extends from the lower quartile to the upper quartile, with total box length corresponding to the interquartile range (IQR). The box is divided by the median. The whiskers correspond to parameter values lying in a range of  $\pm 1.5 \times \text{IQR}$ ; circles denote values beyond these limits. The size of the notch indicates the accuracy of the median, with half the notch width equalling  $1.7 \times \text{IQR} \times \sqrt{n}$ , where  $n$  is the number of single values (samples). Where the notches of two boxes do not overlap, the medians are considered significantly different.

## 3 Results and discussion

### 3.1 Bulk density

Figure 2 shows the bulk density values ( $Da$ ) as box plots, grouped by the type of procedure. It shows that the values obtained for samples only extracted or only prepared by the individual laboratories (LRR or RLR) were not significantly different from those extracted, prepared and analyzed by the reference laboratory (RRR), i.e., the notches overlap. The same applies when comparing procedures RRL and LLL, i.e., when samples were extracted and prepared by the reference laboratory, the values were similar to those obtained when each laboratory performed all tasks, as long as the analysis was done by the laboratory in question. However, clearly different results were obtained depending on whether the samples were analyzed by the individual laboratories or by the reference laboratory, as can be seen by comparing RRR with LLL or RRL. In the latter case, the individual laboratories only analyzed the samples.

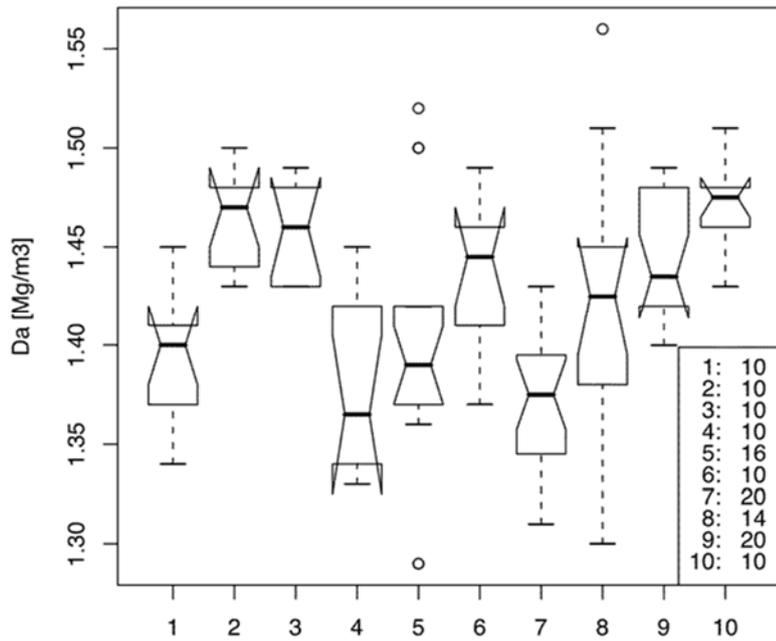


**Figure 2** | Values obtained for bulk density ( $Da$ ), grouped according to the procedure used (see Table 1). Key shows number of samples per box.

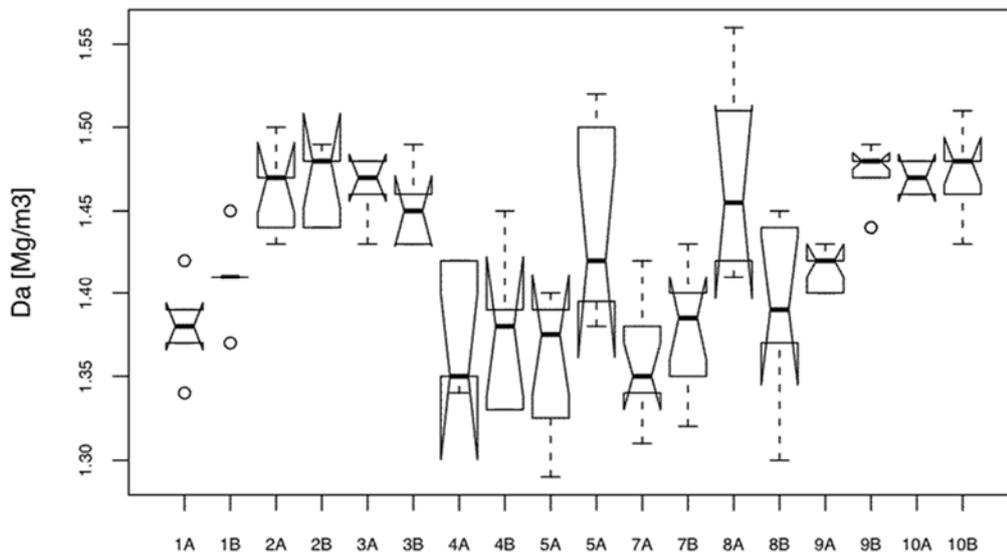
Thus, measured bulk density values ( $Da$ ) appeared to be influenced predominantly by the analysis phase. Therefore, the values obtained by all laboratories, the reference laboratory included, when following procedure 1 were directly compared (Figure 3). It was possible to group the laboratories into those with small values (laboratories 1, 4, 5, 7), those with medium values (6, 8, 9) and those with large values (2, 3, 10). The variation in the values obtained was astonishingly heterogeneous, especially between, for example, laboratory 8 (high variability) and laboratories 2 and 5 (low variability) (Figure 3).

The differences between laboratories cannot be explained by the analytical methods used by the different laboratories. Different ways of handling standard analytical procedures may be the reason for the observed differences.

The bulk density values for sampling areas A and B were significantly different only for laboratories 1 and 9 (Figure 4). In this case, the variability was markedly larger for laboratories 5 and 8 than for the other laboratories.



**Figure 3** | Bulk density ( $D_a$ ) determined by laboratories 1–10 according to procedure 1 (see Table 1). Key shows number of samples per box.

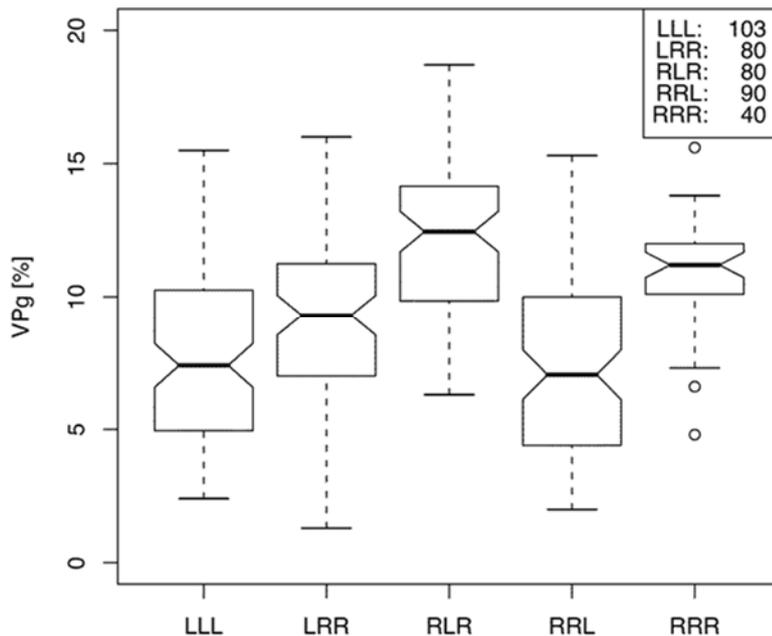


**Figure 4** | Values of bulk density ( $D_a$ ) determined according to procedure 1 (see Table 1) and grouped according to the laboratory (1–10) and the sampling area (A, B).

### 3.2 Macropore volume

Figure 5 shows the values of macropore volume ( $VP_g$ ) at 60 hPa as box plots, grouped by the type of procedure. The values obtained for samples prepared by the individual laboratories (RLR) were greater than those for samples extracted, prepared and analyzed by the reference laboratory (RRR), i.e., the notches are just touching. When the soil samples were only extracted by the individual laboratories (LRR), the values of macropore volume were significantly smaller than those for samples extracted, prepared and analyzed by the reference laboratory (RRR), i.e., the notches do not overlap (Figure 5). When the individual laboratories only analyzed the samples (RRL), again significantly different values were obtained compared with RRR.

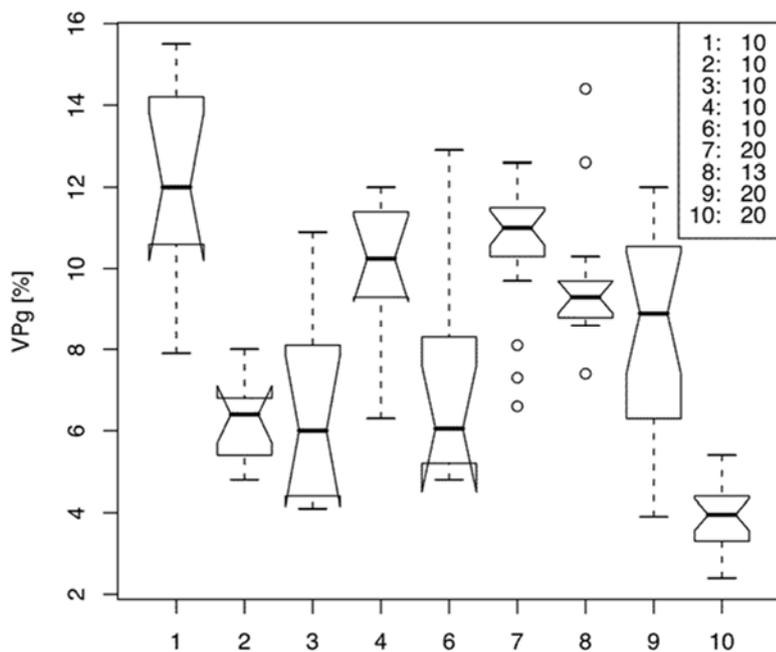
Furthermore, extraction and preparation of samples by the reference laboratory did not lead to significantly different values, as can be seen by comparing the values obtained for LLL and RRL.



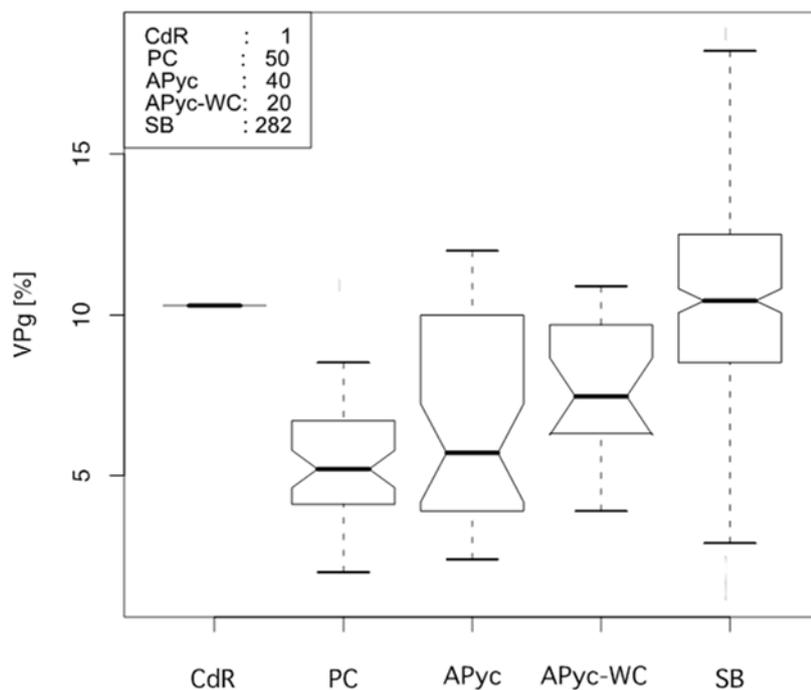
**Figure 5** | Values of macropore volume ( $VP_g$ ) determined at 60 hPa, grouped according to the procedure used (see Table 1). Key shows number of samples per box.

The values of macropore volume at 60 hPa were influenced particularly by the analysis phase. Therefore, the values obtained by all laboratories, the reference laboratory included, when following procedure 1 were directly compared (Figure 6). The values obtained could be divided into four groups: laboratory 10 determined by far the smallest values, rather small values were measured by laboratories 2, 3 and 6, rather large values by laboratories 8 and 9, and the largest values were reported by laboratories 1, 4 and 7. Some laboratories showed small variability in the results (2, 7, 8, 10) and some rather large variability (1, 3, 6, 9).

Grouping the values of macropore volume at 60 hPa according to the methods used (Figure 7) showed that using a pressure cell or air pycnometer for the analysis of macropore volume resulted in values of around 5%. Subtracting the water content at 60 hPa from the water content at saturation (calculated based on total pore volume or on the volume of solid soil substance determined by air pycnometer) tended to give higher macropore volume values than measuring directly with an air pycnometer at 60 hPa, although the differences were not significant. When determined with the sandbox method, the macropore volume values were significantly larger, with a median around 10% and single values ranging from a minimum of 1% to a maximum of 18% (Figure 7). The single value obtained from the shrinkage curve fit within the sandbox values.

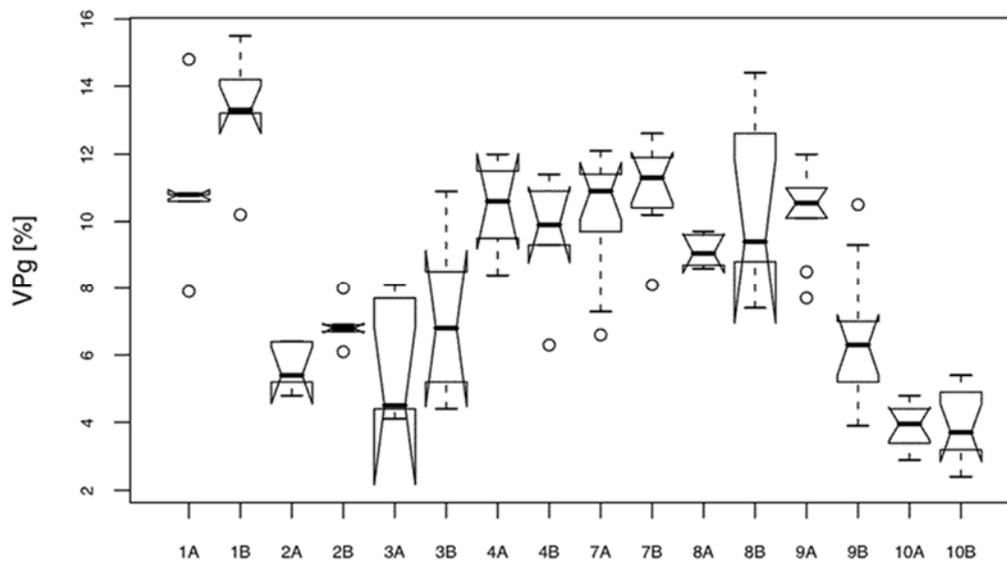


**Figure 6** | Macropore volume (*VPg*) determined at 60 hPa according to procedure 1 (see Table 1). Laboratories 1–8 used one method, laboratories 9 and 10 two methods. Key shows number of samples per box.



**Figure 7** | Macropore volume (*VPg*) determined at 60 hPa with procedure 1 (see Table 1), grouped according to the analytical methods used: CdR = shrinkage curve, PC = pressure cell, APyc = air pycnometer, APyc – WC = water content at saturation (total pore volume or volume of solid soil substance, determined with air pycnometer) minus water content at 60 hPa, SB = sandbox. Key shows number of samples per box.

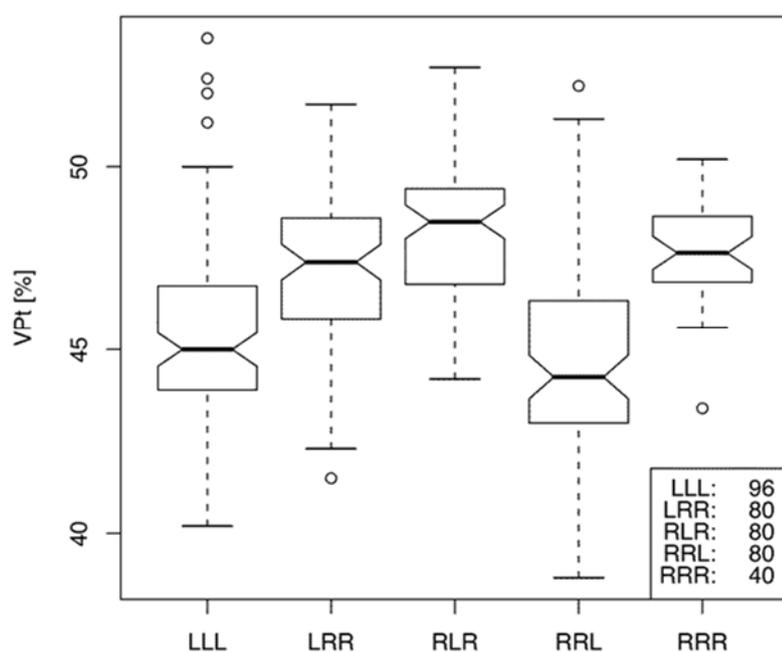
The values of macropore volume obtained for soil samples taken from sampling areas A and B were significantly different for laboratories 1, 2 and 9 (Figure 8). In some situations, the variability in macropore volume was different for the two sampling areas, i.e., was larger either for area A (2A) or B (1B, 8B, 9B, 10B).



**Figure 8** | Macropore volume (VPg) determined at 60 hPa according to procedure 1 (see Table 1) and grouped according to laboratory (1–10) and sampling area (A, B).

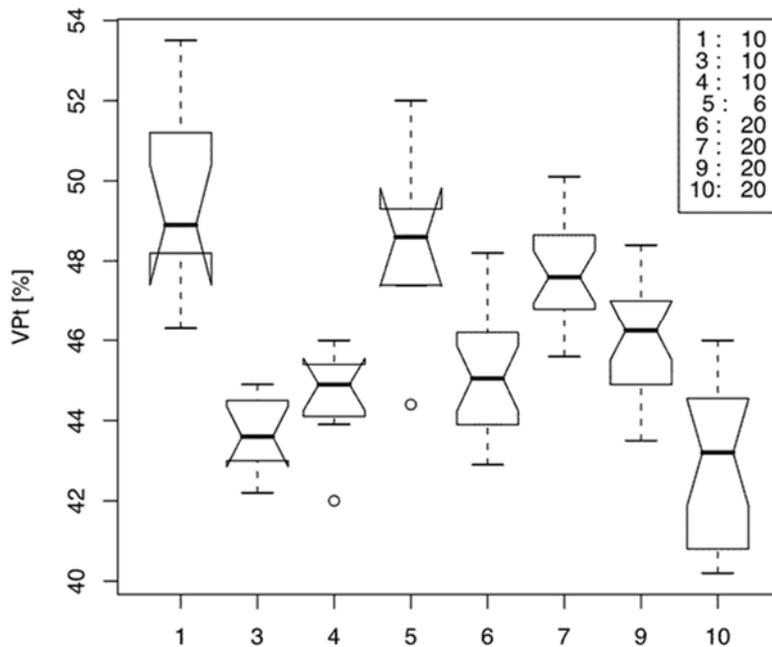
### 3.3 Total pore volume

Figure 9 shows the values of total pore volume ( $VP_t$ ) as box plots, grouped according to the type of procedure. The values obtained for samples only extracted by the individual laboratories (LRR) or only prepared by these laboratories (RLR) were not significantly different from those for samples extracted, prepared and analyzed by the reference laboratory (RRR), i.e., the notches overlap. The same conclusion was reached on comparing the two procedures RRL and LLL, i.e., even when samples were extracted and prepared by the reference laboratory, this did not lead to significantly different values as long as the same individual laboratory did the analysis (Figure 9). However, analysis by the individual laboratories or by the reference laboratory resulted in clearly different results, as can be seen by comparing RRR with LLL or RRL. In the latter case, the individual laboratories only analyzed the samples, whereas the reference laboratory extracted and prepared the soil samples.



**Figure 9** | Total pore volume ( $VP_t$ ), grouped according to the procedure used (see Table 1). Key shows number of samples per box.

The values obtained for total pore volume were influenced most by the analysis phase. Therefore, the values obtained by all laboratories, the reference laboratory included, when following procedure 1 were compared directly (Figure 10). The medians corresponded to total pore volumes between 43 and 49%. The range of single values extended from less than 40% to greater than 53%. It was possible to group the laboratories into those with small values (3, 10), those with medium values (4, 6, 9) and those with large values (1, 5, 7).

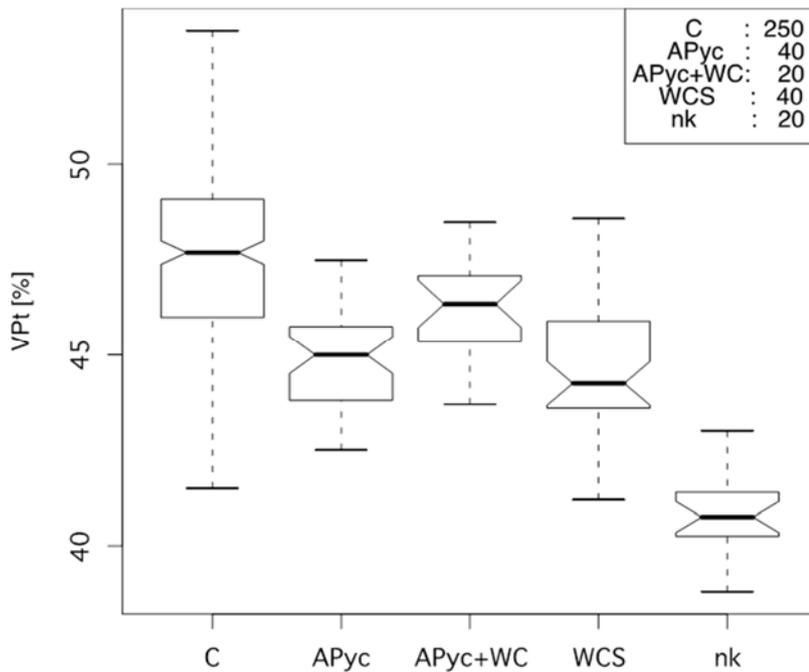


**Figure 10** | Total pore volume ( $VPt$ ) determined according to procedure 1 (see Table 1) and grouped according to laboratory (1–10). Laboratories 6, 9 and 10 used more than one method. Key shows number of samples per box.

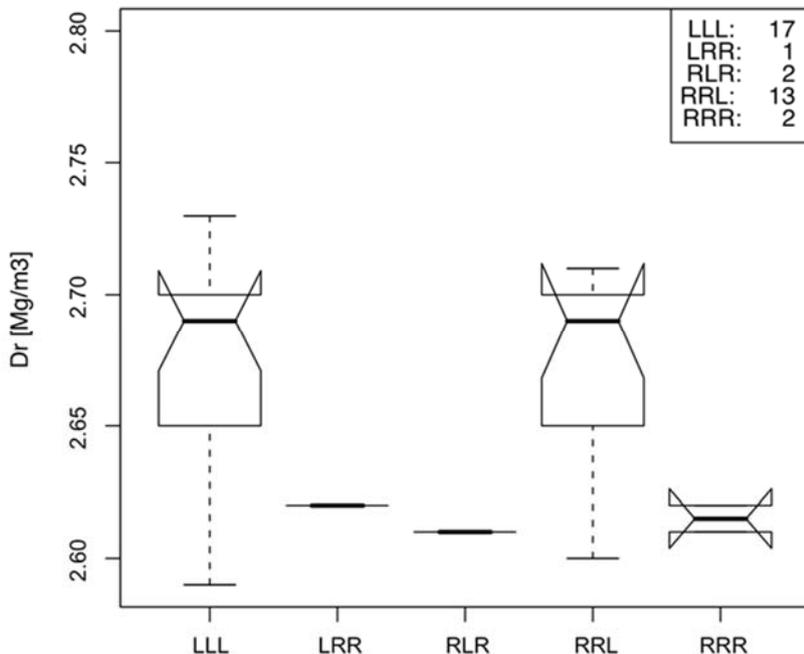
Grouping the values of total pore volume according to the methods used (Figure 11) showed that the method of determining water content at saturation tended to give the smallest values, although these were not significantly different from the larger values obtained with the water pycnometer. Adding water content at 60 hPa, obtained by air pycnometer, to the pore volume at 60 hPa tended to result in greater values of total pore volume than measuring directly by air pycnometer at 60 hPa, although the differences were not significant. Calculating total pore volume from bulk density and particle density was the prevalent method and gave the largest values, significantly greater than those obtained with the other methods (Figure 11).

### 3.4 Particle density

Figure 12 shows the values of particle density ( $D_r$ ) as box plots, grouped by the type of procedure. The values obtained for samples only extracted by the individual laboratories (LRR) or only prepared by these laboratories (RLR) were not significantly different from those for samples extracted, prepared and analyzed by the reference laboratory (RRR). Correspondingly, comparison of the two procedures RRL and LLL showed that having samples extracted and prepared by the reference laboratory did not lead to significantly different values, as long as the analysis was done by the individual laboratory. However, analysis by the individual laboratories or by the reference laboratory resulted in clearly different results, as can be seen by comparing RRR with LLL or RRL. In the latter case, the individual laboratories only analyzed the samples.

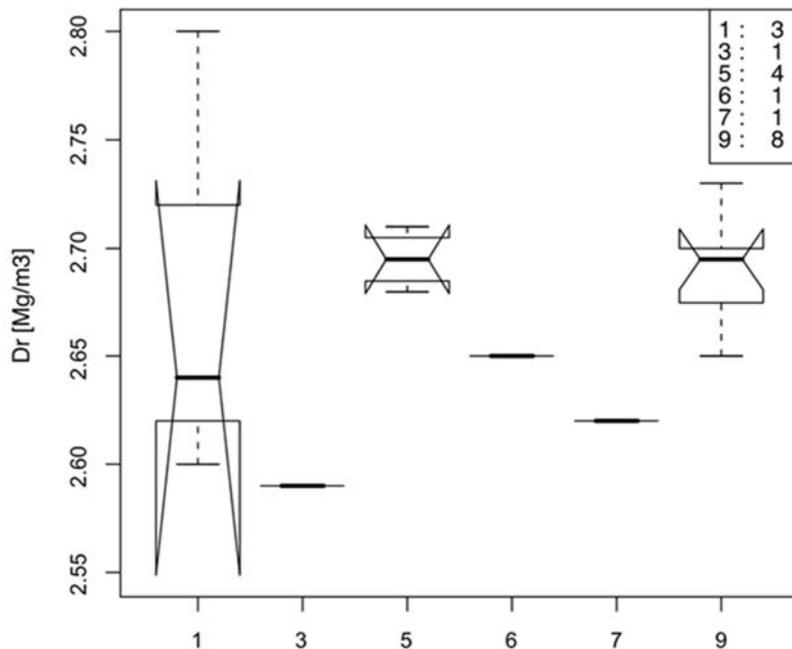


**Figure 11** | Total pore volume (*VPt*) determined according to procedure 1 (see Table 1), grouped according to the analytical methods used: C = calculation based on particle and bulk density, APyc = air pycnometer, APyc + WC = air-filled pore space at 60 hPa (solid/liquid soil substance determined with air pycnometer) plus water content at 60 hPa, WCS = water content at saturation, nk = not known. Key shows number of samples per box.



**Figure 12** | Values of particle density (*Dr*), grouped according to the procedure used (see Table 1). Key shows number of samples per box.

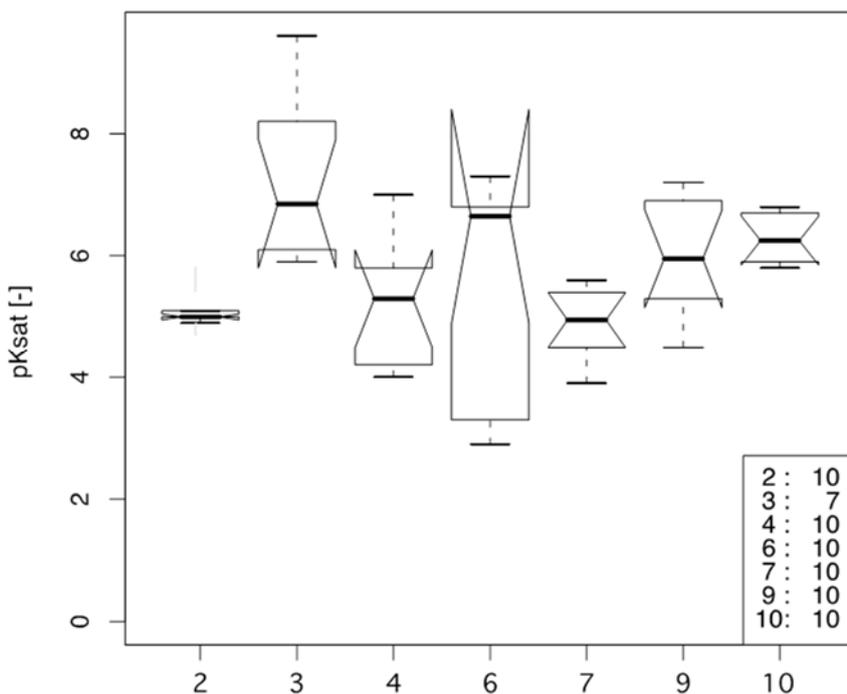
The values obtained for particle density (*Dr*) were particularly influenced by the analysis phase. Therefore, the values obtained by all laboratories, the reference laboratory included, when following procedure 1 were compared directly (Figure 13). Only laboratories 1, 5 and 6 analyzed more than one sample. The large notch of laboratory 1 includes the medians of the values obtained by laboratories 5 and 6, so there is no significant difference (Figure 13).



**Figure 13** | Particle density ( $D_r$ ) determined according to procedure 1 (see Table 1) and grouped according to laboratory. Key shows number of samples per box.

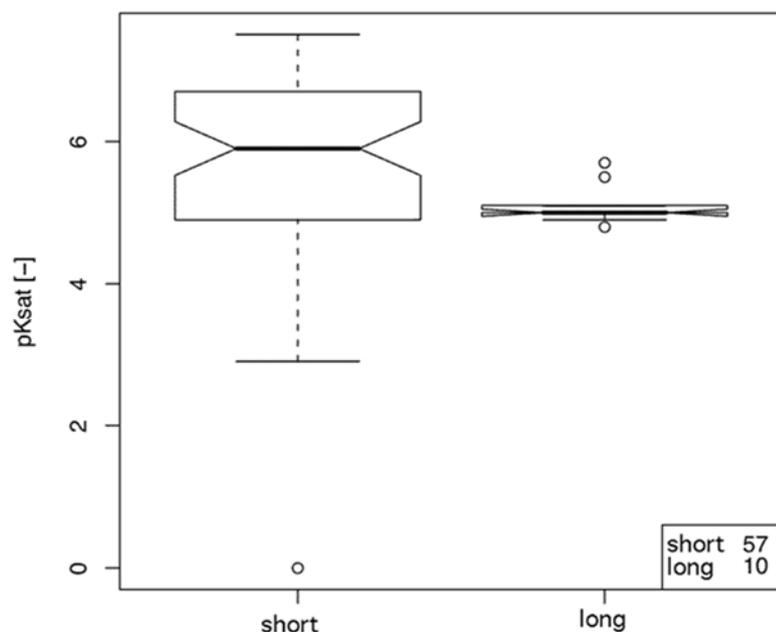
### 3.5 Saturated hydraulic conductivity

Because methods and sample sizes for determining saturated hydraulic conductivity ( $K_{sat}$ ) were not compatible, samples could not be exchanged between the individual laboratories and the reference laboratory. Therefore, only procedure 1 was possible, i.e., the samples were taken, prepared and analyzed by the same laboratory (LLL or RRR). Figure 14 shows the values of saturated hydraulic conductivity measured by the different laboratories. The range of single values of hydraulic conductivity at saturation, expressed as  $pK_{sat}$ , extended from 3 to 8. The medians of  $pK_{sat}$  obtained by the laboratories ranged from 5 to 6.5. A noteworthy finding was the size of the notches, about  $pK_{sat} = 0.1$  for laboratory 2 and 1.5 for laboratory 6.



**Figure 14** | Saturated hydraulic conductivity expressed as  $pK_{sat}$  and determined according to procedure 1 (see Table 1), grouped according to laboratory. Key shows number of samples per box.

Only laboratory 2 used the falling head method. Furthermore, it used samples with a length of 100 mm, at least twice as long as the samples used by the other laboratories. Therefore, grouping the values of hydraulic conductivity at saturation according to the method or the length of the samples gave the same picture (Figure 15). Surprisingly, the longer soil samples exhibited smaller variability than the shorter samples.



**Figure 15** | Saturated hydraulic conductivity expressed as  $pK_{sat}$  and determined following procedure 1 (see Table 1), grouped according to sample length: short = 39–50 mm, long = 100 mm. Key shows number of samples per box.

## 4 Summary

### 4.1 Bulk density

The procedures and methods used for extraction and preparation of samples did not significantly alter the measured values of bulk density. However, the procedures and methods used by different laboratories for analysis of samples resulted in significantly different values. The descriptions of the analytical methods in the different laboratory instruction manuals cannot explain this finding. Therefore, there must be unreported details of handling soil samples or specific ways of implementing standard analytical procedures that have so far not been identified.

Small-scale variations in the soil being sampled were detectable and may have partly contributed to the uncertainty. However, in this interlaboratory comparison, the influence of extracting, preparing and especially analyzing soil samples was greater than the soil variability within the sampling areas.

### 4.2 Macropore volume

The procedures and methods used for extracting and preparing samples resulted in values of macropore volume at 60 hPa that were only slightly different. In contrast, the procedures and methods used for analyzing samples led to significantly different values. As with bulk density, the methodological information in the different laboratory instruction manuals cannot explain this finding. There must be other details of handling soil samples or implementing analytical procedures not uncovered so far.

### 4.3 Total pore volume

The procedures and methods used for extraction and preparation of samples did not significantly alter the measured values of total pore volume. The influence of different methods for determining the total pore volume of soil samples could not precisely be assessed, because the laboratories used three different analytical approaches: (i) determining the mass at saturation and after drying, (ii) calculation based on particle density and bulk density, and (iii) determining the volume of the solid soil substance by air pycnometer.

For the accuracy of method (i), the weight at saturation is crucial because it is quite difficult to ensure complete saturation before and during weighing. Method (ii) is prone to errors because the calculation is based on the ratio of two density values, which in turn is based on two measurements. The accuracy of method (iii) depends on the ratio of the sample volume to the pycnometer volume. Without further information, a concise evaluation of the values measured is not possible.

### 4.4 Saturated hydraulic conductivity

Determining the hydraulic conductivity of short soil samples resulted in much greater uncertainty than using longer soil samples. Values of saturated hydraulic conductivity obtained with soil samples of 100 mm length revealed a very small uncertainty, allowing the same accuracy with markedly fewer samples. The  $pK_{sat}$  values obtained for long soil samples lay within the range of those obtained for short samples but were closer to the lower quartile, indicating better hydraulic conductivity. In contrast, an earlier study found that short soil samples tended to result in higher values of saturated hydraulic conductivity (smaller  $pK_{sat}$  values) than long samples (FaBo, 1998). That finding was explained by a greater probability of continuous macropores in short samples, together with a greater likelihood of fissures between the soil sample and the cylinder wall. Smaller conductivity values, i.e., larger  $pK_{sat}$  values, of short samples as found in this study, may be explained by improper handling during extraction and preparation of soil samples (smeared cross-section surfaces). However, because only procedure 1 was feasible here, no definite explanation is possible.

## 5 Conclusions

The results of this interlaboratory comparison led to the following conclusions:

- (i) When determining bulk density, macropore volume, total pore volume and saturated hydraulic conductivity in soil samples, the values obtained were significantly different when more than one laboratory was involved.
- (ii) The influence of the analysis phase in the laboratory was more pronounced than that of extracting and preparing samples, and much greater than that of soil heterogeneity in the sampling areas.
- (iii) Measured values have to be accompanied by values characterizing the uncertainty caused by the measurement process. This information necessitates a thorough error propagation analysis of the methods and procedures used by a laboratory.
- (iv) The analytical methods for determining these soil physical parameters have to be improved and standardized, particularly that for measuring saturated hydraulic conductivity.
- (v) There is an urgent need for reference samples to check laboratory procedures thoroughly, allowing reproducible measurements of porosity and conductivity for interlaboratory comparisons.

## References

FaBo, 1998. Messung der gesättigten Wasserleitfähigkeit (*K<sub>sat</sub>*) an ‚ungestörten‘ Bodenproben. Interner Bericht, Fachstelle Bodenschutz, Kanton Zürich.

R Development Core Team, 2011. R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. ISBN 3-900051-07-0, [www.R-project.org](http://www.R-project.org)

Swiss Soil Science Society, 2004. Definition und Erfassung von Bodenschadverdichtungen – Positionspapier. Dokument Nr. 13, Bodenkundliche Gesellschaft der Schweiz, Zollikofen.

WRB, 2006. A framework for international classification, correlation and communication. IUSS Working Group, World Reference Base for Soil Resources. World Soil Resources Reports No. 103, FAO, Rome.

## List of figures

- Figure 1** | Left: sampling areas A and B for procedures 1 and 2 by laboratories 1–10. Right: sampling area C for procedures 3 and 4 by reference laboratory 7 ..... 6
- Figure 2** | Values obtained for bulk density ( $D_a$ ), grouped according to the procedure used (see Table 1). Key shows number of samples per box. .... 9
- Figure 3** | Bulk density ( $D_a$ ) determined by laboratories 1–10 according to procedure 1 (see Table 1). Key shows number of samples per box..... 10
- Figure 4** | Values of bulk density ( $D_a$ ) determined according to procedure 1 (see Table 1) and grouped according to the laboratory (1–10) and the sampling area (A, B). .... 10
- Figure 5** | Values of macropore volume ( $VP_g$ ) determined at 60 hPa, grouped according to the procedure used (see Table 1). Key shows number of samples per box. .... 11
- Figure 6** | Macropore volume ( $VP_g$ ) determined at 60 hPa according to procedure 1 (see Table 1). Laboratories 1–8 used one method, laboratories 9 and 10 two methods. Key shows number of samples per box. .... 12
- Figure 7** | Macropore volume ( $VP_g$ ) determined at 60 hPa with procedure 1 (see Table 1), grouped according to the analytical methods used: CdR = shrinkage curve, PC = pressure cell, APyc = air pycnometer, APyc – WC = water content at saturation (total pore volume or volume of solid soil substance, determined with air pycnometer) minus water content at 60 hPa, SB = sandbox. Key shows number of samples per box. .... 12
- Figure 8** | Macropore volume ( $VP_g$ ) determined at 60 hPa according to procedure 1 (see Table 1) and grouped according to laboratory (1–10) and sampling area (A, B). .... 13
- Figure 9** | Total pore volume ( $VP_t$ ), grouped according to the procedure used (see Table 1). Key shows number of samples per box. .... 14
- Figure 10** | Total pore volume ( $VP_t$ ) determined according to procedure 1 (see Table 1) and grouped according to laboratory (1–10). Laboratories 6, 9 and 10 used more than one method. Key shows number of samples per box. .... 15
- Figure 11** | Total pore volume ( $VP_t$ ) determined according to procedure 1 (see Table 1), grouped according to the analytical methods used: C = calculation based on particle and bulk density, APyc = air pycnometer, APyc + WC = air-filled pore space at 60 hPa (solid/liquid soil substance determined with air pycnometer) plus water content at 60 hPa, WCS = water content at saturation, nk = not known. Key shows number of samples per box. .... 16
- Figure 12** | Values of particle density ( $D_r$ ), grouped according to the procedure used (see Table 1). Key shows number of samples per box. .... 16
- Figure 13** | Particle density ( $D_r$ ) determined according to procedure 1 (see Table 1) and grouped according to laboratory. Key shows number of samples per box. .... 17
- Figure 14** | Saturated hydraulic conductivity expressed as  $pK_{sat}$  and determined according to procedure 1 (see Table 1), grouped according to laboratory. Key shows number of samples per box. .... 17
- Figure 15** | Saturated hydraulic conductivity expressed as  $pK_{sat}$  and determined following procedure 1 (see Table 1), grouped according to sample length: short = 39–50 mm, long = 100 mm. Key shows number of samples per box. .... 18