Reproducibility of the Wet Part of the Soil Water Retention Curve : A European Interlaboratory Comparison

Benjamin Guillaume[1], Hanane Aroui Boukbida[2], Gerben Bakker[3], Andrzej Bieganowski[4], Yves Brostaux[1], Wim Cornelis[5], Wolfgang Durner[6], Christian Hartmann[2], Bo V. Iversen[7], Mathieu Javaux[8], Joachim Ingwersen[13], Krzysztof Lamorski[4], Axel Lamparter[9], András Makó[10], Ana María Mingot Soriano[11], Ingmar Messing[11], Attila Nemes[12], Alexandre Pomes-Bordedebat[1], Martine van der Ploeg[3], Tobias Weber Karl David[13], Lutz Weihermüller[14], Joost Wellens[1], and Aurore Degré[1]

[1]Uliège - Gembloux Agro-Bio Tech, TERRA Teaching and Research Centre, Passage des Déportés 2, 5030 Gembloux, Belgium
[2]Instrumentation, Moyens Analytiques, observatoire en Géophysique et Océanographie (UAR IMAGO), Institut de Recherche pour le Développement (IRD)
[3]Wageningen University and Research, Netherlands
[4]Institute of Agrophysics, Polish Academy of Sciences, Poland
[5]Ghent University, Belgium
[6]Technische Universität Braunschweig, Germany
[7]Department of Agroecology, Aarhus University, Denmark
[8]UCLouvain, Earth and Life Institute, Belgium
[9]Federal Institute for Geosciences and Natural Resources, Germany
[10]Department of Soil Physics and Water Management, Institute for Soil Sciences, Centre for Agricultural Research, Herman Ottó street 15, 1022 Budapest, Hungary
[11]Swedish University of Agricultural Sciences, Sweden
[12]Norwegian Institute of Bioeconomy Research, Norway
[13]Institute of Soil Science and Land Evaluation, University of Hohenheim, Germany
[14]Agrosphere Institute IBG-3, Forschungszentrum Jülich GmbH, Germany

Correspondence: Benjamin Guillaume (benjamin.guillaume@uliege.be)

Abstract. The soil water retention curve (SWRC) is a key soil property required for predicting basic hydrological processes. SWRC is often obtained in laboratory with non-harmonized methods. Moreover, procedures associated to each method are not standardized. This can induce a lack of reproducibility between laboratories using different methods and procedures or using the same methods with different procedures. The goal of this study was to estimate the inter/intralaboratory variability of the

5   measurement of the wet part (from 10 to 300 hPa) of the SWRC. An interlaboratory comparison was conducted between 14 laboratories, using artificially constructed, porous and structured samples as references. The bulk densities of samples were different at the very beginning of the experiment. This resulted in a variability of retention properties between the samples, which was estimated by a linear mixed model with a "sample" random effect. Our estimate of inter/intralaboratory variability was therefore not affected by intrinsic differences between samples. The greatest portion of the differences in the measure-

10   ment of SWRCs was due to interlaboratory variability. The intralaboratory variability was highly variable depending on the laboratory. Some laboratories successfully reproduced the same SWRC on the same sample, while others did not. The mean intralaboratory variability over all laboratories was smaller than the mean interlaboratory variability. A possible explanation

for these results is that all laboratories used slightly different methods and procedures. We believe that this result may be of
great importance regarding the quality of SWRC databases built by pooling SWRCs obtained in different laboratories. The
15 quality of pedotransfer functions or maps that might be derived is probably hampered by this inter-/intralaboratory variability.
The way forward is that measurement procedures of the SWRC need to be harmonized and standardized.

1   Introduction

Soil hydraulic properties control important hydrological processes such as infiltration, runoff and evaporation (Assouline,
2021). The soil water retention curve (SWRC) is a soil specific hydraulic property that represents the relationship between
20 the *matric potential* and the *water content* of the soil (Hopmans, 2019). The matric potential represents the energy state of
water in soil, induced by physicochemical interactions between soil particles and water molecules (Luo et al., 2022). These
physicochemical interactions are divided into capillary forces dominating at the wet part of the SWRC and adsorptive forces
dominating at the dry part (Tuller et al., 1999; Tuller and Or, 2005). The wet part of the SWRC is considerably influenced by
the soil pore network on a micrometer scale, which is affected by the so-called "soil structure". This highlights that the SWRC
25 and hydrophysical behaviour of soils can be modified by management practices that influence its structure.

SWRCs are difficult, expensive and time consuming to obtain. SWRC data are therefore limited in space and time. The
SWRC is obtained from the joint determination of a series soil matric potential and soil water content. Since the wet part of
the SWRC is mostly determined by the distribution and connectivity of the largest pores ($> 1\mu$m), it must be measured *in situ*
or in the laboratory on undisturbed soil samples. Soil water content can be measured by direct (gravimetric) method in the
30 laboratory. To obtain matric potential, most laboratory methods impose a target matric potential on an undisturbed soil sample
using an apparatus (Sand box, Sand/Kaolinite box, Suction plate, Pressure plate) (Klute, 1986; Dane and Hopmans, 2002;
Mosquera et al., 2021). The sample is drained until its matric potential reaches equilibrium with the target matric potential.
The SWRC can also be obtained via inverse modelling from an outflow experiment (One step outflow, Multi step outflow)
(Hopmans et al., 2002). The SWRC can also be obtained by simultaneously measuring the water content and matric potential
35 (with a tensiometer) of a soil sample evaporating in the free air and sealed at the bottom. Evaporation experiments also allow
the soil hydraulic conductivity curve to be obtained simultaneously with the SWRC (Peters and Durner, 2008). The Kelvin
equation may also be used to relate the relative humidity of the air in a closed chamber in vapor equilibrium with the soil water
into a matric potential (Dew Point Hygrometer) (Gee et al., 1992).

Each method has its own accuracy and range of measurable matric potential. The determination of the SWRC over the full
40 tension range (between saturation and wilting point or beyond) requires a combination of these methods. The comparison of
these methods shows that they can lead to systematically different SWRCs for samples from the same soil (Bittelli and Flury,
2009; Schelle et al., 2013; Mosquera et al., 2021). The sources of errors are various and may relate to procedural factors,
such as sample size (Ghanbarian et al., 2015; Silva et al., 2018). Pressure plates suffer from apparent hydrostatic equilibria
leading to overestimations of the water content, especially in the dry part of the SWRC (Madsen et al., 1986; Gee et al., 2002;
45 Cresswell et al., 2008; Bittelli and Flury, 2009; Solone et al., 2012; Hunt et al., 2013; Schelle et al., 2013; de Jong van Lier

et al., 2019). In order to prevent hydrostatic non-equilibrium, contact materials between the sample and the porous plate are frequently used to improve the contact (Klute, 1986; Reynolds and Topp, 1993). The effects of these procedural aspects are not clearly established (Gee et al., 2002; Gubiani et al., 2013).

The methods that have been used, up to date, to measure the SWRC are different between laboratories, leading to non-harmonized datasets. Also, procedures for the same method differ from one laboratory to another. As a consequence, most SWRC databases that are used to create pedotransfer functions and maps pool non harmonized data from different laboratories (Wösten et al., 1999; Nemes et al., 2001; Weynants et al., 2013; Tóth et al., 2015, 2017). It is argued that an important source of uncertainty of pedotransfer functions comes from the uncertainty of measured input data and that the standardization of experimental protocols could significantly enhance their quality (Vereecken et al., 2010; Van Looy et al., 2017).

The Soil Program on Hydro Physics via International Engagement (SOPHIE), an independent initiative gathering European stakeholders in the field of soil hydrophysics, focuses on the harmonization and standardization of soil hydrophysics properties through international collaboration. No interlaboratory comparison for the measurement of the SWRC has yet been carried out. This is partly due to the fact that real soil samples can neither be transported from one laboratory to another nor measured several times without affecting their SWRC. SOPHIE works towards innovating measurement techniques, development of reference samples and organizing interlaboratory comparisons, starting with the SWRC.

This paper presents the results of first SOPHIE interlaboratory comparison for the measurement of the wet part (from 10 to 300 hPa) of the SWRC on reference samples. Fourteen laboratories participated in this study using their typical routine measurement methods and protocols. Four research questions were addressed:

1. What are the "intralaboratory" variabilities of the 14 participating laboratories?

2. What is the "interlaboratory" variability of the 14 participating laboratories?

3. Are the reference samples different between each other?

4. Are the reference samples affected by time, measurements and/or transport between laboratories?

2   Materials and Methods

2.1   The reference sample

Each reference sample was composed by a mixture of 180 g of glass beads (0.250 mm < x < 0.500 mm), 20 g of pure air dry Portland cement and 35g of tap water. Once homogenized, the mixture was filled into a 100 cm^3 (5 cm height/ 5 cm diameter) stainless steel ring by frequently tapping it on a table to ensure that it was uniformly packed. The ring was closed at the bottom by a lid. Any excess material on the top was removed with a spatula. Each sample was allowed to cure for 72h at room temperature. The bottom lid was subsequently replaced with a Eijkelkamp nylon cloth supported by a rubber band. The sample, with the ring, the cloth and the rubber, was weighted. The empty ring, the cloth and the rubber were previously weighted separately.
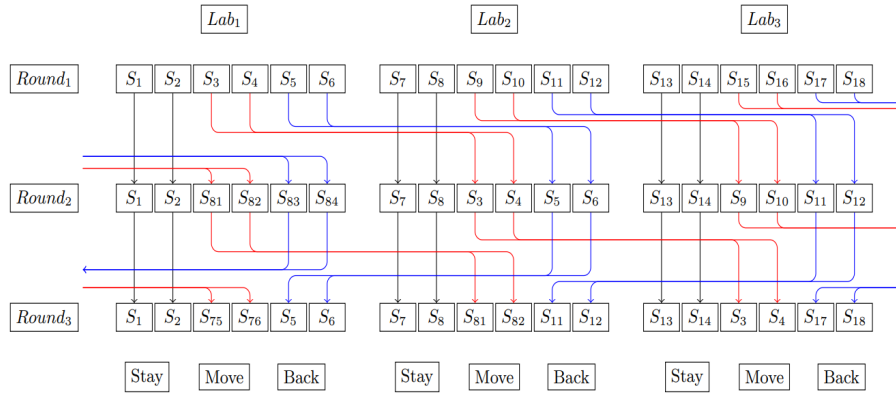
Figure 1. Example of sample exchange scheme of the ring test. Black arrows = STAY, red arrows = MOVE, blue arrows = BACK.

2.2 The ring test

The ring test was organized in 14 soil physics laboratories. An example of a reference sample was sent to each laboratory alongside the material needed to construct five other samples. A total of 84 reference samples were constructed. The ring

80 test consisted of three successive rounds of SWRC measurements. At each round, the mass of each sample was measured at 10 hPa (pF = $\log_{10}$(hPa) = 1.00), 50 hPa (pF =1.70), 100 hPa (pF = 2.00) and 300 hPa (pF = 2.48) after saturation for 48h. Equilibration times were 5 days at 10 hPa, 7 days at 50 hPa, 10 days at 100 hPa and 15 days at 300 hPa. Finally, samples were weighed after drying for 72 hours in an oven drying at 60°C. Gravimetric water content (wc in g.g^{-1}) was calculated by the ratio of fresh over dry masses. The six samples from each laboratory were divided into three exchange modalities (Fig. 1); 2

85 samples were kept by the same laboratory all along the 3 rounds of measurements ("STAY"), two samples were sent to different laboratories between rounds ("MOVE") and the last two samples were sent to a different laboratory for the second round but were sent back to the original laboratory for the third round ("BACK"). This scheme was designed to estimate intralaboratory and interlaboratory variability as well as the effect of sample transfer between laboratories.

2.3 The data analysis

90 The final data set consisted of 250 SWRCs. Two curves were missing. Since each SWRC was composed of four successive measurement points whose relative value may depend on the previous point, the data were not independent. Statistical analyses were then based on parameter values of fitted functions. The measured wet part of SWRCs was adjusted to a linear function with $log_{10}(hPa) = pF$ as the independent variable and was used to model our dataset (Eq. 1).

$$wc_i = \beta_0 + (\beta_1 * (pF_i - 1)) + \epsilon_i \tag{1}$$

95 Water content (wc_i) was linearly expressed as a function of pF values (pF_i = 1.00, 1.70, 2.00, 2.48). The $pF_i - 1$ was intended to set the first retention point at the intercept. The index i represented the ith data point. β_0 and β_1 represented the mean

intercept and the slope over all data. The term ϵ_i represented the residuals. The next step was to adjust a single linear model to each SWRC (Eq. 2).

$$wc_{in} = \underbrace{\beta_0 + z_{0n}}_{\text{varying intercept}} + \underbrace{((\beta_1 + z_{1n}) * (pF_i - 1))}_{\text{varying slope}} + \epsilon_{in} \tag{2}$$

100 The n index represented the nth SWRC. Depending on the modeled SWRC, intercepts (z_{0n}) and slopes (z_{1n}) were allowed to vary around a general intercept (β_0) and slope (β_1). This type of model refers to a linear mixed (effect) model.

The purpose of this study was also to investigate the interlaboratory variability as well as the differences between samples. Another linear mixed (effects) model was used to consider the by sample and by laboratory variability using adjustment terms called "random effects". The first random effect, u_{0j} and u_{1j} respectively adjusted β_0 and β_1 depending on the analysing

105 laboratory j ($j \in [1, ..., 14]$). The other random effect, v_{0k} and v_{1k} respectively adjusted β_0 and β_1 depending on the sample k ($k \in [1, ..., 84]$). This mixed effects model was described by the Eq. (3).

$$wc_{ijk} = \underbrace{\beta_0 + u_{0j} + v_{0k}}_{\text{varying intercept}} + \underbrace{((\beta_1 + u_{1j} + v_{1k}) * (pF_i - 1))}_{\text{varying slope}} + \epsilon_{ijk} \tag{3}$$

Finally, the effect of sample changes between round 1 and round 3 on the intercept (w_0) and the slope (w_1) was modeled

110 through a "fixed effect" covariate. The covariate depended on a dummy variable associated to the round number; for the 1st round, $round_i = -0.5$ and for the 3rd round, $round_i = 0.5$. This later model (Eq. 4) was applied only to data associated with the "BACK" samples and "STAY" samples to avoid laboratory effects. The results were compared to determine whether the differences in measurements between rounds 1 and 3 were due to transport or differences caused by wear of the samples not related to transport.

115 $$wc_{ik} = \underbrace{\beta_0 + v_{0k} + w_0 * round_i}_{\text{varying intercept}} + \underbrace{((\beta_1 + v_{1k} + w_1 * round_i) * (pF_i - 1))}_{\text{varying slope}} + \epsilon_{ik} \tag{4}$$

All parameters from each models were estimated using Bayesian statistics. Posterior distributions were sampled with a Markov Chain Monte Carlo (MCMC) algorithm implemented in C++ through an R package called "RStan" (Carpenter et al., 2017). Sensitivity analyses of priors and validations of models were also carried out. Inference was based on Bayes factors and Bayesian Credible Intervals of posterior distributions. More details are available in the Supplementary Materials (doi :).

120 # 3 Results

3.1 Procedures of laboratories

Each laboratory received the same procedure to measure the SWRC. However, it allowed some freedom and some laboratories did not perfectly implement it. For instance, laboratory 8 dried the samples at 100°C instead of 60°C. Hence, laboratories used slightly different procedures as shown in Table 1. Laboratories mostly used the Sand Box (SB) at 10, 50 and 100 hPa. At 300

Table 1. Specificity of the participating laboratories in terms of device used (SB = Sand Box, SKB = Sand/Kaolinite Box, SP = Suction Plate, PP = Pressure Plate), contact material, cap on the samples during equilibration periods, reference level used with respect to the sample at which the pressure was set, unit correction, plate cleaning procedure, saturation procedure and dry weight measurement procedures.

Lab	Device				Contact material	Cap	Pressure sample ref	Correction cm to hPa	Plate clean	Saturation		Cooling after drying
	10 hPa	50 hPa	100 hPa	300 hPa						Water type	Water level	
1	SB	SB	SB	SP	spheriglass 3000	/	middle	/	water + brush	/	/	/
2	SB	SB	SB	SP	quartz meal	no	middle	no	no	demineralized	45 mm	dessicator
3	SB	SB	SB	PP	no	no	middle	yes	H2O2 + water	demineralized	25 mm	dessicator
4	SP	SP	PP	PP	milled sand	no	middle/unknown	no	water	demineralized	47 mm	NA
5	SP	SP	SP	SP	filter paper	no	middle	yes	no	tap	49 mm	dessicator
6	SB	SB	SB	PP	kaolinite on filter paper	no	middle	yes	light HClO solution	demineralized	49 mm	no cooling
7	SB	SKB	SKB	SKB	no	yes	middle	yes	no	distilled	50 mm	dessicator
8	SB	SB	PP	PP	no	yes	bottom	no	water + brush	deaerated	50 mm	no cooling
9	SB	SB	SB	PP	loamy soil	no	middle	yes	water + brush	tap	40 mm	dessicator
10	SP	SP	SP	SP	no	yes	bottom	no	water	distilled	45 mm	NA
11	SP	SP	SP	PP	filter paper	yes	middle	no	water	demineralized	/	NA
12	PP	PP	PP	PP	no	no	middle	yes	water	demineralized	40 mm	in the oven
13	SB	SB	SP	SP	sand	no	bottom	no	water	demineralized	50 mm	in the oven
14	SB	SB	SB	PP	no	no	middle/bottom	no	tap water + brush	demineralized	45 mm	NA
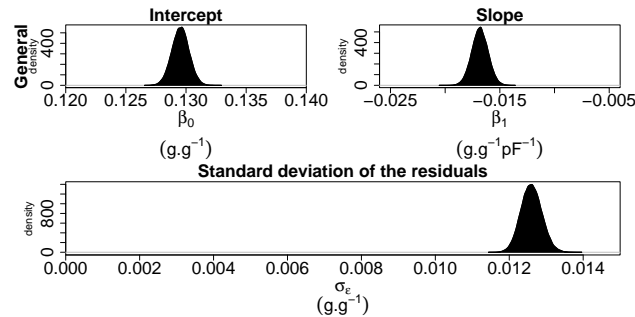
Figure 2. Densities of the posterior probability distribution of the general intercept, β_0, the general slope, β_1, and the standard deviation of the residuals, σ_ϵ.

125   hPa, the Suction Plate (SP) and the Pressure plate (PP) were dominating. Lab 7 was the only one to use the Sand/Kaolinite Box (SKB).

3.2 The simple linear model : SWRCs are very variable

The simple linear regression (Eq. 1) with the pF as predictor was used to model the data set. The posterior probability distribution of the general intercept (β_0), slope (β_1) and the standard deviation of the residuals (σ_ϵ) are shown in Fig. 2. The mean value

130   of σ_ϵ was fairly high (0.0126 g.g^{-1}). Indeed, as shown in Fig. 3, the variability of measured SWRCs was large (spreading of the curves). The following steps were devoted to explaining the origin of this variability.
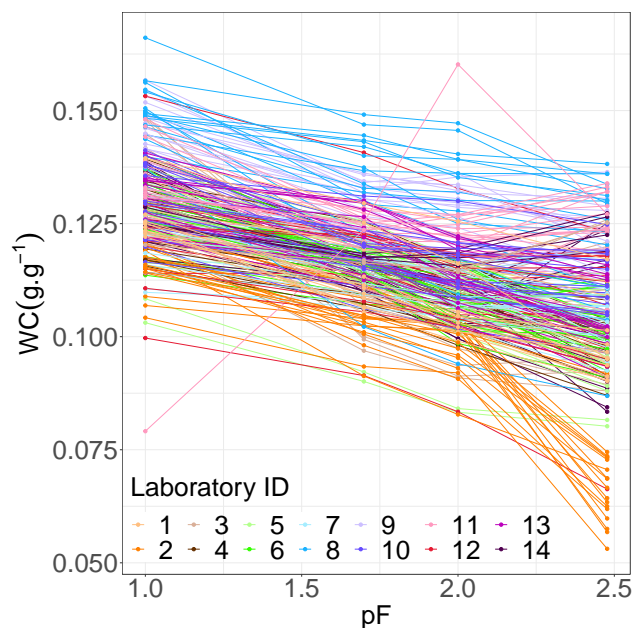
Figure 3. SWRCs of the entire data set expressed with gravimetric (g.g^{-1}) water contents. One colour represents one laboratory. This colour code is kept constant throughout the paper.

3.3 A linear model for each SWRC to estimate the intralaboratory variability

The next step was to model a single linear regression for each SWRC (Eq. 2). The posterior probability distribution of the general intercept (β_0), slope (β_1), the individual intercept (z_{0n}) and slope (z_{1n}) and the standard deviation of the residuals (σ_ϵ)

135 are shown in Fig. 4. The intercept (z_{0n}) and slope (z_{1n}) parameters are different for each individual SWRC. These individual parameters explain the variability that exists between all SWRC. Hence, the mean value of σ_ϵ presented in Fig. 4 decreased by approximately 60 % compared to the previous model (Fig. 2) and now only represents a fitting error introduced by the choice of modeling SWRCs by linear regressions.

From these results, one can determine the standard deviation of z_{0n} and z_{1n} for each of the "STAY" samples (between the

140 3 rounds). As each laboratory measured two "STAY" samples, an estimate of the intralaboratory variability of each laboratory can be made by pooling the density estimates of the standard deviation of the two samples (Fig. 5). The estimate of the mean intralaboratory standard deviation of all laboratories pooled together (Fig. 5 bottom row) was 0.00533 g g^{-1} (95% Credible intervals (CrI) 0.00018-0.01138 g g^{-1}) for the intercept and 0.00519 g g^{-1}pF^{-1} (95% CrI 0.00038-0.01068 g g^{-1}pF^{-1}) for the slope (Table A1). Figure 5 (top row) also shows that the intralaboratory variability was quite different depending on the

145 laboratory. Some laboratories succeeded in repeating similar SWRCs results on a same sample while others failed.
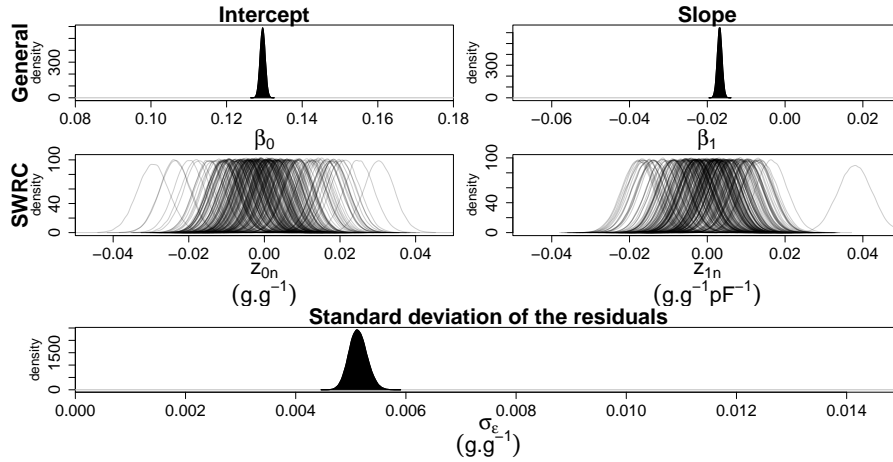
Figure 4. Densities of the posterior probability distribution of the general intercept, β_0, the varying intercept, z_{0n}, the general slope, β_1, the varying slope, z_{1n}, and the standard deviation of the residuals, σ_ϵ.
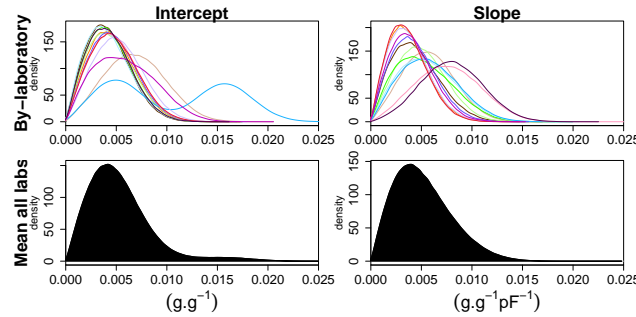
Figure 5. Densities of the posterior probability distribution of the standard deviation of the two "STAY" samples of each laboratory (top row) and all laboratories together (bottom row).

3.4   What is the interlaboratory and sample variability?

Although all laboratories were given the same procedure to build the reference samples, the conditions under which they were constructed differed between laboratories. Hence, the bulk density of samples at the beginning of the experiment was variable depending on the laboratory that constructed the sample (Table A2). Indeed, the difference between the mean bulk density

150   of samples constructed by the lab 1 and lab 14 was 0,1573 g.cm^{-3}. Hence, the later linear mixed (effect) model was used to estimate the interlaboratory variability on the SWRC considering the differences between samples (Eq. 3). Densities of the posterior probability distribution of the general intercept (β_0) and slope (β_1), the random effect of laboratory on the intercept (u_{0j}) and slope (u_{1j}), the random effect of sample on the intercept (v_{0k}) and slope (v_{1k}) and the standard deviation of the residuals (σ_ϵ) are shown in Fig. 6. The mean value of σ_ϵ presented in Fig. 6 decreased by approximately 40 % compared to the

155   simple linear model (Fig. 1). Indeed, a part of the variability between SWRCs has been explained by sample and laboratory
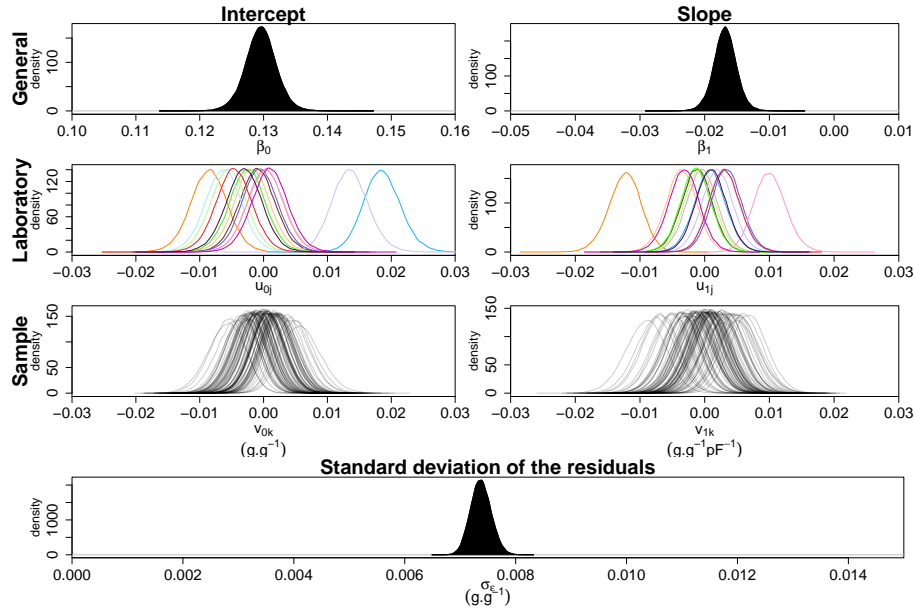
Figure 6. Densities of the posterior probability distribution of the general intercept (β_0), the random effect of laboratory on the intercept (u_{0j}), the random effect of sample on the intercept (v_{0k}), the general slope (β_1), the random effect of laboratory on the slope (u_{1j}), the random effect of sample on the slope (v_{1k}) and the standard deviation of the residuals (σ_ϵ).

random effects. Parameter values of the laboratory random effects (u_{0j} and u_{1j}) show how SWRCs systematically deviate depending on the analysing laboratory. Differences between samples were also estimated with parameter values of the samples random effects (v_{0j} and v_{1j}). The wider dispersion of the laboratory random effect parameters indicates that the analysing laboratory explained a larger proportion of the overall variance than the analysed sample. Indeed, on the intercept, the mean

160   laboratory random effect standard deviation (σ_{u0}) was 0.00872 g g^{-1} while it was 0.00350 g g^{-1} for the sample random effect (σ_{v0}). The same observation applies to the slope with a mean standard deviation of 0.00602 g g^{-1}pF^{-1} for the laboratory random effect (σ_{u1}) and 0.00451 g g^{-1}pF^{-1} for the sample random effect (σ_{v1}). The mean laboratory random effect standard deviations on the intercept and slope values (σ_{u0} and σ_{u1}) represent an estimation of the interlaboratory variability.

3.5   Do the samples change between rounds?

165   In order to assess the effect of possible sample changes on the SWRCs measurements, a last model was separately fitted to the data from "BACK" and "STAY" samples (Eq. 4). The Bayes factor indicated that the predicted data of "BACK" samples were 46.60 times more probable under the model that takes the round effect into account than the model without the round effect. Moreover, the 95% credible interval of the posterior probability distribution of the "round" effect (Fig. 7) laid outside 0 for the intercept (w_{0b}) and the slope (w_{1b}). Therefore, "BACK" samples changed between round 1 and round 3. On the other hand, the

170   Bayes factor indicated that the predicted data of "STAY" samples were 4348 times less probable under the model that takes
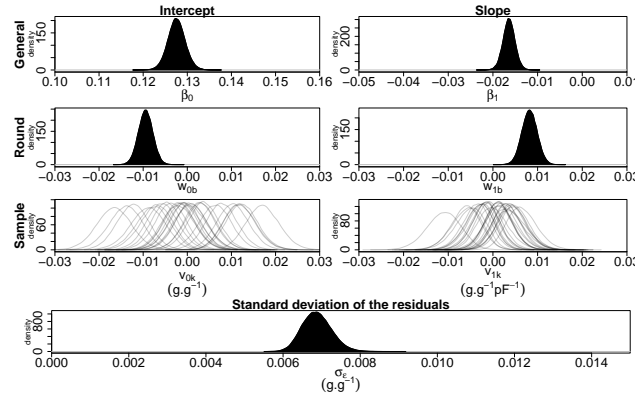
Figure 7. Densities of the posterior probability distribution of the general intercept (β_0), the fixed effect of the transport on the intercept (w_{0b}), the random effect of sample on the intercept (v_{0k}), the general slope (β_1), the fixed effect of the transport on the slope (w_{1b}), the random effect of sample on the slope (v_{1k}) and the standard deviation of the residuals (σ_ϵ). The model is applied to the "BACK" data for the 1st and the 3rd rounds.
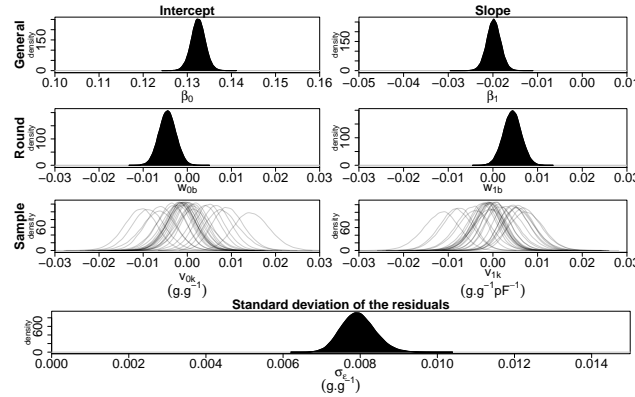
Figure 8. Densities of the posterior probability distribution of the general intercept (β_0), the fixed effect of the transport on the intercept (w_{0s}), the random effect of sample on the intercept (v_{0k}), the general slope (β_1), the fixed effect of the transport on the slope (w_{1s}), the random effect of sample on the slope (v_{1k}) and the standard deviation of the residuals (σ_ϵ). The model is applied to the "STAY" data for the 1st and the 3rd rounds.

the round effect into account than the model without the round effect. Nevertheless, the 95% credible interval of the posterior probability distribution of the "round" effect (Fig. 8) laid outside 0 for the intercept (w_{0s}) and the slope (w_{1s}). Therefore, for the "STAY" samples, the Bayes factor and 95% credible interval yielded to opposite conclusions. Also, the dry mass of "STAY" samples increased between round 2 and 3 (p = 0.016*) which was not the case with "BACK" samples (p = 0.199 ns). The round effect on "STAY" samples is discussed in section 4.3.

175

3.6 Increasing SWCRs

57 of the 250 measured SWRCs showed an increase in water content between at least two increasing suction steps (Fig. 3). Whatever the origin, this increase of water content is not physically acceptable. It appeared that the occurrence of these anomalies depended on the analysing laboratory, with some having no anomalies and others having a large number of occurrences. Indeed, laboratories 3, 11 and 14 together accounted for 35 of the 57 anomalies recorded (Table A3). Moreover, this anomaly has happened more than once for some samples such as for the samples 1, 2, 11, 15, 56, 61, 62, 63, 65 and 66. The samples 15, 63, 65 and 66 showed this anomaly in two different laboratories. Also, it occurred more often the drier the sample was. There were 55 occurrences between 100 and 300 hPa while there were only 9 between 50 and 100 hPa and 3 between 10 and 50 hPa. For some SWRCs, there was more than one occurrence.

4 Discussion

4.1 interlaboratory variability

This study confirms that there are systematic differences in the measurement of SWRCs depending on the laboratory (Fig. 6). This is true even for laboratories using similar devices (eg. lab 6 vs 9). These systematic differences in the measurement of SWRCs attributed to laboratories resulted in a large interlaboratory variability. The portion of variability attributed to differences between laboratories was larger than the portion of variability attributed to intrinsic differences between samples. This is concerning since it was shown, through the comparison of the bulk densities (Table A2), that the samples were different even at the very beginning of the experiment. From saturation to drying, all laboratories used slightly different procedures that can be at the origin of this interlaboratory variability (Table 1). The identification of the aspects of the procedures that influence SWRCs measurements is challenging since these were not studied in isolation. This is a multidimensional problem that remains beyond the scope of this article. Nonetheless, an attempt is made to hypothesize the effect of some of these procedural aspects. It should also be mentioned that the true value of the SWRC was unknown. Laboratories were compared according to their relative position with respect to the others and not against a fixed target value.

Differences between laboratories are unlikely to be associated with differences in the analysed samples. The intrinsic differences between samples was considered by the model (Eq. 3) using the sample random effect. Indeed, there was no correlation between the intercept parameter of each laboratory and the average bulk density of the samples analysed by each laboratory (r = -0.0078).

A first possible source of this interlaboratory variability could be attributed to the different devices used. To our knowledge, no study has yet attempted to compare SWRCs obtained with SB, SKB, SP or PP. Nonetheless, Schelle et al. (2013) found that SWRCs measured with SP were less reproducible (wider spread) than those measured with the evaporation method in the pF range 0-2.5. They also found that, for sandy soils, water contents are systematically smaller for SWRCs obtained with SP than with the evaporation method.
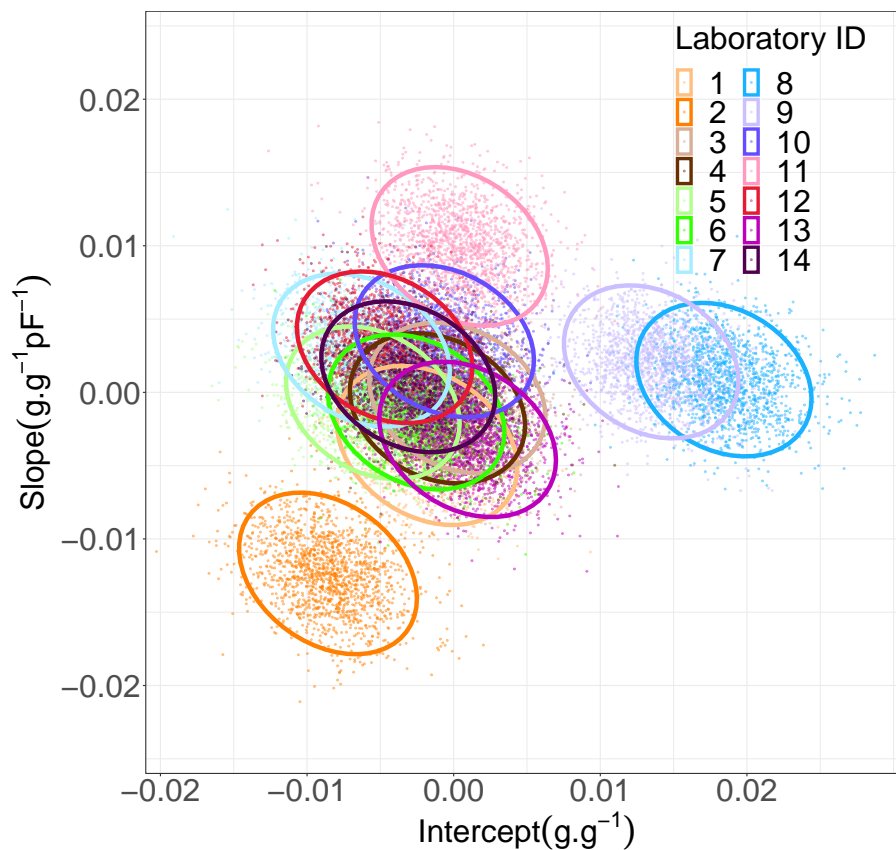
Figure 9. Joint posterior probability distribution of the laboratory random effect on the intercept (u_{0j}) and slope (u_{1j}).

Moreover, all laboratories used two different devices between 10 and 300 hPa, except labs 5, 10 and 12 that kept the same device for each pressure step. Changing from one device to another (from suction to pressure system) may affect the measurement of the SWRC. Water in non-uniform pores with non-continuous liquid phase between the top and the bottom of 210 the sample might be subject to different pressure gradient when extraction is via air pressure or via water suction.

The procedures for the dry mass measurement of the samples may also have played a role in the observed differences. Indeed, the estimation of the intercept parameter of laboratory 8, which dried the samples at 100°C, was higher than the ones from the other laboratories, which dried the samples at 60°C (Fig. 6). This suggests that the dry masses measured by laboratory 8 were lower than those measured by the other laboratories.

215 Another possibility to explain differences is the way laboratories maintained hydraulic contact between the draining porous media and the sample, enabling water to be released from the sample until hydrostatic equilibrium is reached. When the draining porous media was rigid (eg. ceramic) some laboratories used a "contact material" to improve the hydraulic contact

(Table 1). From this study, it seems that the use of contact materials does not systematically ensure hydraulic contact between samples and porous plates. In laboratory 11, the filter paper failed to improve hydraulic contact.

220   Nevertheless, the use of contact materials may sometimes be useful when considering laboratories using the same devices. Hence, it appears that the use of filter paper by laboratory 5 resulted in more water being released (more negative slope) than laboratory 10, which did not use any contact material but used the same devices (Fig. 9). Gubiani et al. (2013) also found that filter paper allowed more water to be released than polyester fabric and synthetic knitwear at 5000 and 15000 hPa with the PP. The use of kaolinite by lab 6 and loamy soil by lab 9 as contact material seems to yield in more water being released between

225   100 and 300 hPa than laboratories 3 and 14 that did not used any contact material but have used the same devices (results not shown). However, when looking at the whole curve (from 10 to 300 hPa) the effect of kaolinite or loamy soil was negligible (Fig.9). Gee et al. (2002) also found kaolinite ineffective in speeding equilibrium (or increasing hydraulic conductance), with inconsistent effects, at 15 000 hPa. Further work should be done to determine which contact materials is useful depending on the specific situation.

230   An option to check if the hydrostatic equilibrium is achieved is to connect the porous drainage medium to a graduated cylinder and monitor the water level/weight. If the water level/weight does not increase, hydrostatic equilibrium is considered to be achieved. This setup has to be sealed in order to ensure that there is no evaporation. The advantage of such a system is that one does not need to assume the equilibration time a priori. To our knowledge, this setup was used by laboratory number 2 and 8. However, it is still possible, with this setup, that the hydraulic contact is broken and the flow of water is stopped before

235   hydrostatic equilibrium is reached which refers to as an apparent hydrostatic equilibrium.

It should also be mentioned that with devices using a hanging water column as suction regulation system, the applied suction is usually expressed in cm of water column. However, 1 cm of water column is not equal to 1 hPa but 0.98 hPa. This error of 2% is usually overlooked when units are transformed (cf. Table 1). This may constitute a small part of the variability between laboratories and calls for harmonization of units.

240   In addition, the reference level compared to the sample at which the suction is applied varies between laboratories and devices used. Some laboratories applied the prescribed suctions to the bottom of the samples while others applied it to the middle (cf. Table 1). Laboratories that applied suction to the bottom of the samples systematically applied 2.5 cm more suction than those that applied it to the middle.

There might be other procedural aspects that can be responsible for these differences between laboratories (saturation pro-

245   cedure, porous plate maintenance during the experiment, means of preventing air leakages and evaporation, maintenance of the ceramics and the sandboxes, weighting procedure, maintenance of the scales, etc.)(Table 1). Other sources of uncertainty may relate to the lab environment (temperature and humidity). Big errors can be avoided by a quality check of the results. To our knowledge, only one laboratory used a reference sample to control the quality of their SWRCs measurements as a standard operating procedure.
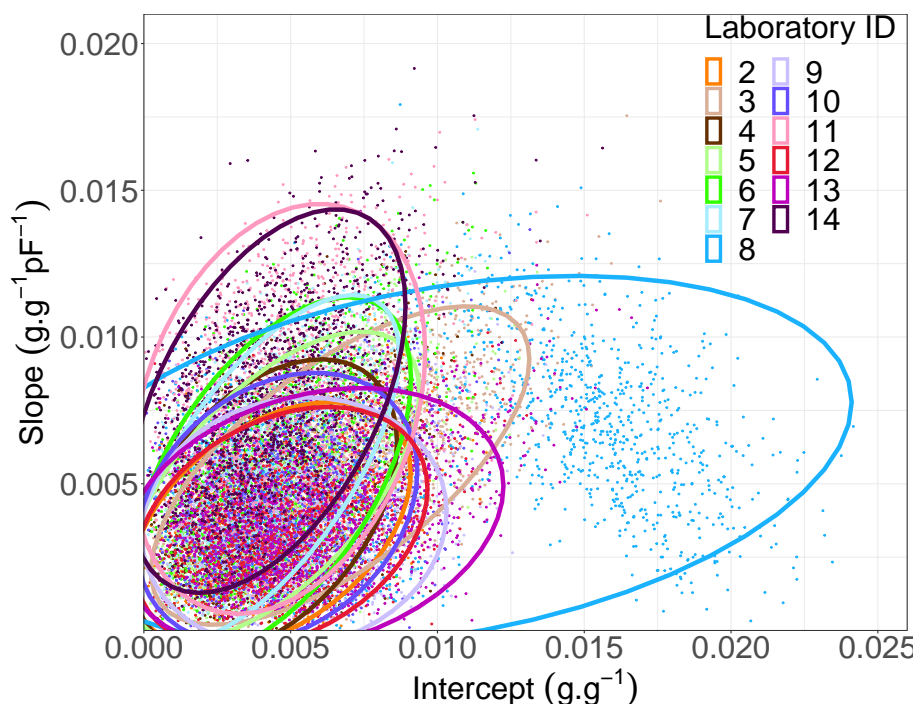
Figure 10. Joint posterior probability distribution of intralaboratory standard deviations on the intercept (z_{0n}) and slope (z_{1n}).

4.2 intralaboratory variability

250 Some laboratories successfully reproduced SWRCs of a same ("STAY") sample while others failed (Fig. 10). Nevertheless, the estimate of the mean intralaboratory variability over all laboratories was smaller than the mean interlaboratory variability, but was more uncertain since it was drawn from less samples and since the intralaboratory variability was quite different between laboratories. Obviously, this variability can partly be attributed to the different methods and procedures that existed between laboratories that were discussed above. Some procedures ensured fairly good repeatability of results while others did not.

255 The two laboratories with the greatest intralaboratory variability on the slope (cf. Fig. 5 & 10: red and dark blue curves) were also among those with the most anomalies (cf. Table A3: lab 11 and 14). Concerning laboratory 8 (cf. Fig. 5: cyan curve), the bimodal shape of the intralaboratory variability on the intercept shows that for one sample the variability was high while it was low for the second. This bimodality clearly indicates that the estimation of individual intralaboratory variabilities are rather

260 uncertain as they are only based on two samples measured with only three repetitions. This calls for further trials on reference
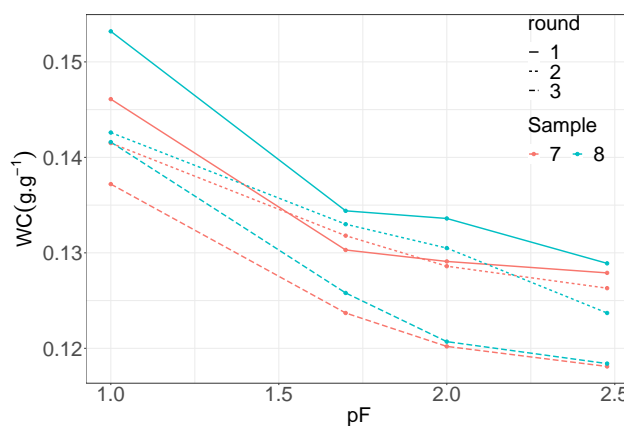
Figure 11. SWRCs of the "STAY" samples measured by lab 9.

samples to obtain a more reliable estimate of intralaboratory variability. Nevertheless, this provides an insight into the way forward to improve data quality management in soil physics laboratories.

4.3 Effect of repeated measurements and/or transport on the samples

It appears that there was a slight effect of the transfer between laboratories on the samples. The values of w_{0b} and w_{1b} indicate that SWRCs of "BACK" samples globally have a smaller intercept with a flatter slope after being transported (Fig. 7). This pattern might indicate a shift to more small pores. A possible explanation for these changes in porosity is the calcium carbonation of the cement. This reaction forms $CaCO_3$ precipitates inside the pore network inducing a shift of the pore size distribution towards smaller pores, a decrease of the total porosity, pore clogging and a loss of pore connectivity in cement based materials (Šavija and Luković, 2016; Auroy et al., 2015). This hypothesis is also motivated by the fact that the dry masses of the samples increased significantly between rounds 2 and 3 for the samples "STAY" and the dry masses did not decrease significantly for the samples "BACK" even if losses of materials were reported by the laboratories. Indeed, Houst (1993) estimated that the carbonation induced increase in bulk density (due to CO_2 fixation) from a non carbonated to a fully carbonated cement paste was 1.60 to 2.03 g.cm^3. However, the actual contribution of this phenomenon to changes in the retention properties of the reference samples is difficult to estimate, as the degree of carbonation was influenced by environmental factors (CO_2 concentration, air humidity, water content of the cement, etc.) which have not been controlled. Nevertheless, this significant transport effect could have led to an overestimation of the interlaboratory variability, as a part of the variability of the SWRC measurements can be attributed to sample changes. The use of cement to construct such reference samples should certainly be avoided in the future.

Although it was not significant in general for "STAY" samples, some laboratories still seems to report sample changes between rounds. The changes followed the same patterns as for "BACK" samples, which were significant it that case (Fig. 8). Indeed, for "STAY" samples of laboratories 2, 3, 4, 6, 9, 10, 11, 12 and 13 it seems that the water content at 10 hPa (pF 1) systematically decreased with time. This was particularly visible for lab 9 (Fig. 11). Without a doubt, the origin of the changes

of the "STAY" samples can partly be attributed to the same origin as for the "BACK" samples. The degree of changes may therefore be influenced by the way the samples were handled and stored, resulting in less wear for non transported samples. Nevertheless, the wear of the "STAY" samples implies that the estimation of the intralaboratory variability was certainly inflated

285 as it included the variability imputed to sample changes between rounds. It should be mentioned that for some laboratories with the highest "intralaboratory" variability (laboratories 8 and 14), this trend was not visible, indicating that for these laboratories the variability attributed to procedures was probably more important than the variability attributed by sample changes.

4.4 Outliers

Many reasons might be elicited to explain the fact that some SWRCs showed an increase in water content between at least two

290 increasing suction steps (Fig. 3). Obviously, this happened depending on a combination of reasons related to the laboratory but also to the sample being analyzed (Table A3).

A possible reason can be the lack of hydraulic contact between the draining porous media and the sample, preventing water to be released in time from the sample. This is supported by the higher frequency of outliers when the sample was drier, as hydraulic conductivity decreases as the sample dries (Gee et al., 2002). Indeed, there is a possible scenario in which samples

295 may absorb water but may not be able to release it according to the driving (higher) pressure. Measurements in a pressure chamber typically involve placing samples on pre wetted ceramic plates. However, especially when a wet contact material is used, a unsaturated sample may start absorbing water (from the plate and the contact material) and resaturate before the chamber is pressurized. Once the chamber is pressurized the excess of water may not be drained if the hydraulic contact is not well established. Hydraulic contact could have been hampered by the rigid nature and non flat bottom topography of reference

300 samples which did not fit the porous plate or by the use of shrinkable contact materials. When using the pressure plate, it is also possible that a "backflow" of water from the ceramic to the sample may occur between the release of the pressurized air and the disconnection of the sample from the plate (Richards and Ogata, 1961). Nevertheless, increasing SWRC also occurred with sandboxes and sand/kaolinite boxes, where the applied suction was not released when the sample was disconnected.

An other possible explanation is that the mass of the samples probably increased during the measurements due to the

305 inclusion of CO_2 induced by the carbonation of cement, as discussed above. It is therefore possible that the total mass of a sample slightly increased between two suction steps if the mass increase induced by the inclusion of CO_2 was greater than the mass loss due to the release of water (prevented by a lack of hydraulic contact). However, this explanation does not hold for some of the largest increases in mass recorded which may be due to other reasons.

5 Conclusions

310 The objective of this study was to estimate the inter/intralaboratory variability of the measurement of the wet part of the SWRC. An interlaboratory comparison was conduced between 14 laboratories for the first time. Artificially constructed, structured and porous samples were used as references. The bulk densities of the samples were different from the very beginning of the experiment. This induced variable retention properties between samples, which was considered in a linear mixed model by a

"sample" random effect. Systematic differences in the measurement of SWRCs attributed to laboratories resulted in a large interlaboratory variability. The variability explained by the differences between laboratories was more important than the variability explained by intrinsic differences between samples. The intralaboratory variability was laboratory dependent. The mean intralaboratory variability over all laboratories was approximately 45% smaller on the intercept and 15% smaller on the slope than the mean interlaboratory variability (Table A1). Samples slightly changed during the interlaboratory comparison which has probably led to overestimates of the intra/interlaboratory variabilities. The intra/interlaboratory variability can also be attributed to the different methods and procedures followed by each laboratory that could not be fully standardized.

This variability needs to be reduced by improving and standardizing procedures and harmonizing methods. Ideally, all laboratories should endorse a unique Standard Operational Procedure for the same method and methods should be harmonized between each other. Improving and standardizing procedures requires a full assessment of the effect of each step of the procedures on the final SWRC measurement. Harmonization of methods can be achieved with interlaboratory comparisons. Nevertheless, this requires that the reference samples remain stable over time, which was not the case in this study. Further work is needed to design new reference samples (e.g. with clays, sintered glass beads or ceramics) that can be used by laboratory as internal and external quality control. Since procedures and methods could have an impact on the final measurement of the SWRC, the transparency of the procedures and methods used in SWRCs datasets should be ensured. These recommendations aim to contribute to the improvement of knowledge of hydrological processes and to the consistency of databases built on multiple laboratories' inputs, such as those used to derive the most widely used pedotransfer functions. Without such an effort, pedotransfer functions and large scale maps of soil properties will keep carrying unknown levels of uncertainty and bias.

Code and data availability. Data and R code will be available on an online and open access repository linked from the manuscript through a DOI.

Table A1. Summary table of interlaboratory and intralaboratory variability results.

Lab	Laboratory random effect on the intercept, u_0 (g.g^{-1})			Laboratory random effect on the slope, u_1 (g.g^{-1}.pF^{-1})			Intralab SD on the intercept (g.g^{-1})			Intralab SD on the slope (g.g^{-1}.pF^{-1})		
	Mean	95% CRI		**Mean**	95% CRI		**Mean**	95% CRI		**Mean**	95% CRI	
1	**-0,00184**	-0,00780	0,00396	**-0,00363**	-0,00851	0,00110	**/**	/	/	**/**	/	/
2	**-0,00855**	-0,01450	-0,00278	**-0,01237**	-0,01770	-0,00762	**0,00460**	0,00047	0,00877	**0,00377**	0,00032	0,00752
3	**0,00021**	-0,00570	0,00611	**-0,00044**	-0,00532	0,00428	**0,00689**	0,00129	0,01251	**0,00576**	0,00094	0,01050
4	**-0,00105**	-0,00675	0,00472	**-0,00123**	-0,00608	0,00348	**0,00423**	0,00040	0,00825	**0,00450**	0,00041	0,00897
5	**-0,00552**	-0,01134	0,00014	**-0,00082**	-0,00562	0,00397	**0,00457**	0,00048	0,00898	**0,00505**	0,00064	0,00961
6	**-0,00259**	-0,00850	0,00314	**-0,00138**	-0,00614	0,00328	**0,00435**	0,00046	0,00850	**0,00531**	0,00046	0,01052
7	**-0,00636**	-0,01228	-0,00069	**0,00291**	-0,00181	0,00775	**0,00424**	0,00042	0,00845	**0,00570**	0,00063	0,01095
8	**0,01853**	0,01275	0,02450	**0,00081**	-0,00413	0,00558	**0,01049**	0,00128	0,01986	**0,00584**	0,00065	0,01103
9	**0,01350**	0,00767	0,01949	**0,00198**	-0,00292	0,00683	**0,00524**	0,00077	0,00985	**0,00383**	0,00036	0,00763
10	**-0,00060**	-0,00649	0,00520	**0,00338**	-0,00135	0,00822	**0,00473**	0,00051	0,00896	**0,00430**	0,00045	0,00846
11	**0,00025**	-0,00570	0,00602	**0,01009**	0,00530	0,01516	**0,00483**	0,00064	0,00923	**0,00764**	0,00152	0,01366
12	**-0,00468**	-0,01062	0,00099	**0,00308**	-0,00167	0,00798	**0,00482**	0,00057	0,00924	**0,00370**	0,00030	0,00736
13	**0,00095**	-0,00494	0,00675	**-0,00322**	-0,00809	0,00152	**0,00591**	0,00061	0,01151	**0,00408**	0,00037	0,00807
14	**-0,00310**	-0,00902	0,00249	**0,00100**	-0,00384	0,00578	**0,00438**	0,00049	0,00855	**0,00802**	0,00205	0,01378
Overall SD	**0,00872**	0,00562	0,01367	**0,00602**	0,00370	0,00968	**0,00533**	0,00018	0,01138	**0,00519**	0,00038	0,01068

Appendix A: Supplemental tables

Table A2. Newman and Keuls' groups of populations of samples bulk density according to the laboratory that constructed them. Lab number 15 represents the examples samples provided by UGent.

Lab number	Mean (g.cm^{-3})	SD (g.cm^{-3})	Pop. size	NK Group
1	1.8035	0.0094	5	a
2	1.7781	0.0141	5	b
8	1.7639	0.0494	5	b c
3	1.7551	0.0049	5	b c d
11	1.7540	0.0090	5	b c d
7	1.7528	0.0046	5	b c d
10	1.7425	0.0062	5	c d
12	1.7314	0.0168	5	d
4	1.6948	0.0198	5	e
13	1.6657	0.0291	5	f
5	1.6579	0.0133	5	f
6	1.6574	0.0177	5	f
9	1.6489	0.0056	5	f
14	1.6462	0.0136	5	f
15	1.6359	0.0113	14	f

Table A3. ID of Samples showing increasing SWRCs as function of the analysing laboratory and the round.

Lab	Round 1	Round 2	Round 3
1	/	/	/
2	/	/	/
3	1, 2, 5	1, 2, 39, 40, 41, 42	1, 5, 81, 82
4	/	/	/
5	/	15, 17, 18	/
6	43	/	15, 16
7	56, 57, 59, 60	48, 56	/
8	/	/	/
9	10, 11	/	11
10	/	/	/
11	62, 63, 64, 65	61, 62, 75, 76, 77, 78	9, 61, 62, 65, 66
12	/	65, 66	25, 29, 30
13	/	/	63
14	21	20, 69, 70, 71, 72	23, 28

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