STANDARDIZED METHODS FOR THE PHYSICAL ANALYSIS OF PLANT SUBSTRATES

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Abstract
A set of "reference" and "routine" procedures for the measurement of the physical conditions in plant substrates were tested in seven institutes. The measurements included bulk density, pore volume and volume fractions of water and air at certain moisture potentials (pF-values). The precision within the institutes was found to be good (i.e. the analytical errors of the single analyses were small). The agreement between the institutes still leaves much to be desired. To improve standardization, continued interlaboratory testing is recommended.

Introduction

Consultations with colleagues led to a proposal for standardized procedures for the measurement of physical conditions in plant substrates (Van Dijk, 1976). Two "reference" procedures consist of elaborate measurements of bulk density, pore volume and volume percentages of water and air at pF 1.0, 1.5 and 2.0, one in which the substrates are compressed as done in pots and one without compression, imitating the situation in loosely filled beds or bags. These two procedures are intended particularly for scientific experiments. To meet the requirement for rapid "routine" measurements, in addition, simplified modifications for both procedures are proposed. These include bulk density, pore volume and volume percentages of water and air at pF 1.5 only.

In 1977, these four procedures were tested in seven laboratories in Western Europe with the same nine plant substrates of different composition. Purpose of this test was first of all to establish the precision of the reference methods and the degree of agreement between the laboratories and, secondly, to see whether the routine procedures would turn out to be acceptable simplifications. At the ISHS symposium in Dublin, September 1977, Verdonck et al. (1978) gave a preliminary report of the results available at that time. They concluded that the agreement between the results of the different institutes was very poor. On the other hand, I could show that the precision of the measurements within an institute can be sufficiently high (Van Dijk, 1978).

Verdonck kindly provided me with all the results to which those from another two laboratories were added after the Dublin meeting. The data were subjected to an analysis of variance in the institute's department of statistics. The results are reported below,

The institutes cooperating in this interlaboratory test were:
1. Lab. of Soil Physics, Ghent, Belgium (Verdonck, De Boodt).

2. Inst. for Soil Fertility, Haren, Netherlands (Boekel, Van Dijk).
3. Exp.Sta.for Glasshouse Crops, Naaldwijk, Netherlands (Sonneveld).
4. Lab.Agron.du SAS, Gargenville, France (Verdure).
5. Min.d'Agric., ENITAH, Angers, France (Rivière).
6. The Macaulay Inst.for Soil Research, Aberdeen, U.K. (Robertson, Boggie).
7. MAFF, A.D.A.S., Shardlow, U.K. (through E.W. Johnson).

Two other laboratories only sent results obtained with their own methods which are not discussed here.

Materials and methods

Six substrates were distributed from Ghent by Dr. Verdonck. They were based on peat (G_1 and G_2), pine litter (G_3), bark compost (G_4 and G_5) and vermiculite (G_6). Three potting soils were distributed from Naaldwijk by Mr. Sonneveld, based on mixtures of peat moss and "black peat" (N_1), of peat moss and sedge peat (N_2) and of peat moss and leaf mould (N_3).

Equipment

- metal cylinders, height 50 mm, inner diameter between 70 and 80 mm (content 200-250 cm^3; to be determined exactly),
- press or weights to compress the substrate in the cylinders with a pressure of 100 and 500 grams per cm^2, respectively,
- knife or other utensil to cut away the substrate in the upper cylinder,
- sandbox with drainage system for the determination of the moisture retention at pF 1.0, 1.5 and 2.0 (i.e. suction tension of 10, 31.6 and 100 cm water, respectively); e.g. length × width × height 60 × 30 × 35 cm, filled with a 20 cm layer of fine sand with an air entry value of about 150 cm of water, covered with a nylon cloth. The drain inside the sandbox near the bottom consists of a frame of pvc-tubing, slotted underneath and wrapped in hydrophylic nylon cloth. The outlet is connected with a levelling bottle, adjustable along a graduated stand by means of which a suction tension of 7.5, 29.1 and 97.5 cm, as taken from the sand surface, can be realized. The sandbox has a neatly fitting lid,
- drying oven,
- balance.

Sample condition

The substrate sample is homogenized by carefully mixing without grinding or sieving; only lumps or clods larger than 2 cm are broken. To prevent incomplete saturation caused by difficult moistening, the moisture content expressed in grams per 100 g dry matter should be (and normally is) at least 1.2 times the weight percentage of organic matter (on a dry-matter basis). When the moisture content is lower, water should be added to the substrate until an amount of 1.4 × % organic matter is present, after which the sample should be stored for 24 hours in a closed plastic bag.

Procedures

The two "reference" procedures (C_{ref} with compression of the substrate and L_{ref} *without compression*) and their shorter modifications C_{rout} and L_{rout} are described in detail by Van Dijk (1976) and Verdonck et al. (1978).

All determinations should be carried out at least in duplicate.

Results

Institutes no. 1 to 4 carried out all 4 procedures on all 9 samples. No. 7 only applied procedure C_{ref}. From no. 5 no results of procedure C_{ref} were received, from no. 6 only results of the samples G_1 to G_6 and procedures C_{ref} and L_{ref}. The analysis of variance was therefore concentrated particularly on the results of institutes 1 to 4.

From institutes 2, 3 and 4, data of duplicates were available. The analytical error and coefficient of variation for bulk density and vol. % of water at pF 1.5 within these institutes is given in Table 1. For physical determinations the precision is certainly satisfactory, even for the shorter routine procedures.

Results of the analyses of the variance per institute are given in Table 2 (for the 4 institutes which supplied the complete set of data). Concerning C_{ref} - L_{ref}, it appears that three institutes succeeded in expressing the effect of compression on all four properties very reliably. As to C_{ref} - C_{rout}: only institute 4 succeeded in getting results with the routine procedure which are nowhere significantly different from those obtained with the reference procedure. In this respect the other institutes succeeded only partly. This could be due to a smaller analytical error there for both procedures, but this is not supported by the data in Table 1. Concerning L_{ref} - L_{rout}: now institutes 3 and 4 are successful in getting the same results with both procedures, for institute 1 this is only partly the case whereas no. 2 obtained significantly different results.

To what extent are the methods satisfactory in distinguishing between substrates? (NB Differences in origin of the materials do not necessarily imply large differences in the physical substrate conditions considered here). To visualize the differentiation between substrates, the results of 6 institutes are shown in figure 1 as an example for bulk density and vol. % of water at pF 1.5 as obtained by applying procedure C_{ref}. In general, each institute succeeded in ascertaining significant differences in properties of the substrates, which is nor surprising in view of the precision of the measurements.

Figure 1 also gives a first impression of the agreement in results between the institutes. From the clustering of the institute's numbers it can be seen that the extent of agreement is different for the individual substrates. Table 3 gives the coefficients of variation between all seven institutes, calculated for all 9 substrates separately. The variation is small for pore volume, higher (often too high) for bulk density and vol. % of water at pF 1.5, and highest for vol. % of air at pF 1.5. The latter is not surprising since errors in pore volume and vol. % of water accumulate in vol. % of air. Substrates G_2, N_3 and particularly G_6 (vermiculite) turn out to be troublesome here. Judging from Table 3, in general the agreement between the institutes is indeed rather poor, in sharp contrast with the precision found within the institutes. (Compare, for example, the last column of Table 3 where the coefficients of variation are given as calculated from the duplicates for all 9 substrates obtained in institute 2). This poor agreement appears to be mainly due to differences in level of data obtained per institute and per procedure. Figure 1, for example, shows that with procedure C_{ref}, institute 1 almost always obtained the lowest data for bulk density and vol. % of water whereas those of institute 2 mostly lay in the top of the range. The order of level is not the same for all four procedures but is rather consistent per procedure.

For example, with procedure L_{ref}, institutes 1 and 2 both obtained relatively high values for bulk density.

Finally, analyses of variance per procedure were restricted to institutes 1 to 4 from which complete data were available and omitting vermiculite. The mean values for the remaining 8 substrates, together with their standard deviations, are given in Table 4. In the last column, the range w_4 obtained by applying the multiple range test of Dixon and Massey (1957) is given. Differences between mean values larger than w_4 are significant. Where this is not the case the mean values in Table 4 are connected by a line. It appears that the differences in level often are small and not significant, particularly between 2, 3 and 4.

Discussion

There is no plausible cause for the supposition that the precision, proven to be high in three institutes, even for the shorter "routine" procedures, has been or would be less other institutes. The procedures apparently are precise enough to register physically important differences between the substrates and the effect of compression. In this respect, therefore, the general adoption of the proposed procedures can be recommended. However, for the time being, also when only the "routine data" are required, the reference procedures should be followed as well, in order to ascertain that there are no differences in level of results. That this can be achieved is indicated by the results of some institutes (Table 2).

Well-standardized methods should exclude the possibility of significant differences occurring in the level of results between different laboratories. It is obvious that we have not yet reached that ideal, although the results expressed in Table 4 are encouraging. It may be that the description of the procedures has not been clear enough to avoid differences in interpretation. (Suggestions for improvement are very welcome). The fact, however, that many institutes did not have all required equipment at their disposal and perhaps had to improvise, may well have been the major cause for differences in level. For example, I know that in one institute metal cylinders were used with an inner diameter of 5 cm instead of the prescribed 7-8 cm. I ascertained that a smaller diameter results in a lower bulk density and higher pore volume, which in this case explains at least part of the differences in level.

It is very common experience in ring tests that the procedures have to be described and followed very carefully to achieve a good agreement in results. Another point which has to be stressed here is that the utmost care has to be taken in subsampling. For some substrates widely varying organic matter contents were reported, for example for G_4 from 43 to 62%, for N_3 from 34 to 50%. It is hardly conceivable that this is due only to differences in method of determination. Concluding, I strongly recommend to continue inter-laboratory testing. It would have been a miracle if already after one test with nine samples the ultimate goal had been achieved.

References

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Table 1 - Mean values, analytical errors and coefficients of variation in the determination of bulk density and of volume percent of water at pF 1.5 carried out by three institutes in the same 9 materials.

Inst.			Procedure			
			C_{ref}	C_{rout}	L_{ref}	L_{rout}
2	bulk dens.	Mean	231	232	201	219
		S_{an}	3.2	6.6	3.8	7.7
		v	1.4	2.8	1.9	3.5
	water pF 1.5	Mean	57.7	55.9	49.1	51.5
		S_{an}	0.4	0.6	0.5	0.6
		v	0.7	1.1	1.1	1.2
3	bulk dens.	Mean	219	214	170	166
		S_{an}	6.6	9.4	6.2	3.5
		v	3.0	4.4	3.7	2.1
	water pF 1.5	Mean	55.4	53.2	45.4	44.3
		S_{an}	0.9	0.7	1.6	1.5
		v	1.6	1.3	3.4	3.3
4	bulk dens.	Mean	214	215	170	165
		S_{an}	11.4	4.9	4.9	10.8
		v	5.3	2.3	2.9	6.5
	water pF 1.5	Mean	56.5	55.4	45.4	42.8
		S_{an}	2.3	1.4	2.0	1.2
		v	4.1	2.6	4.4	2.9

Analytical error $S_{an} = \sqrt{\frac{2d^2}{2N}}$; d = difference between duplicates, N = 9

v = coefficient of variation = S_{an} in % of Mean

Table 2 - F-values and level of significance for the differences in results obtained within 4 institutes with the four procedures.

Comparison of procedures		Institutes 1	2	3	4
$C_{ref} - L_{ref}$	Bulk dens.	3.48	61.52***	118.29***	39.99***
	pore vol.	3.12	59.33***	187.08***	31.18***
	water pF 1.5	2.27	103.07***	152.64***	37.45***
	air pF 1.5	0.43	86.52***	209.55***	42.97***
$C_{ref} - C_{rout}$	Bulk dens.	4.52*	0.02	0.87	0.03
	Pore vol.	4.45*	0.15	1.17	0.03
	water pF 1.5	6.16*	4.39*	7.21*	0.34
	air pF 1.5	2.12	2.07	7.51*	0.19
$L_{ref} - L_{rout}$	Bulk dens.	2.32	22.93***	0.70	0.47
	Pore vol.	2.09	21.98***	1.17	0.11
	water pF 1.5	38.15***	8.01**	1.82	2.07
	air pF 1.5	48.69***	12.55**	2.23	1.07

* Probability level 5 %
** " " 1 %
*** " " 0.1 %

Table 3 - Coefficients of variation (S in % of Mean) between all seven laboratories, calculated for all 9 materials separately.

		G_1	G_2	G_3	N_1	N_2	N_3	G_4	G_5	G_6	All[1)] 9
Bulk dens.	C_{ref}	5.0	7.2	9.1	3.2	3.8	5.7	5.0	7.4	18.4	1.4
	C_{rout}	5.4	7.5	6.4	3.9	5.4	9.2	8.8	14.5	25.9	2.8
	L_{ref}	8.7	15.1	9.2	5.6	4.5	11.6	9.5	8.6	27.7	1.9
	L_{rout}	13.0	16.7	13.3	8.3	9.3	15.3	16.7	13.4	31.8	3.5
Pore vol.	C_{ref}	2.6	0.9	1.9	2.4	1.1	4.1	2.2	3.2	1.5	0.2
	C_{rout}	0.8	0.7	0.9	2.0	1.4	3.2	3.9	1.6	1.7	0.4
	L_{ref}	1.1	1.2	1.5	2.4	1.4	3.5	2.3	2.7	1.7	0.2
	L_{rout}	1.1	1.1	1.4	2.3	1.3	3.4	2.9	2.3	1.7	0.5
water pF 1.5	C_{ref}	7.6	10.3	8.5	8.7	7.4	8.2	6.2	8.1	9.4	0.7
	C_{rout}	5.0	11.5	7.2	6.4	6.1	10.1	9.1	8.1	13.8	1.1
	L_{ref}	9.1	14.6	9.2	7.6	4.1	13.1	8.9	8.9	17.1	1.1
	L_{rout}	7.1	17.0	12.6	6.9	7.3	13.5	10.9	5.2	17.2	1.2
air pF 1.5	C_{ref}	19.3	16.0	12.6	20.6	14.2	15.2	9.3	12.1	21.0	1.4
	C_{rout}	9.8	16.7	8.5	18.9	16.5	19.5	19.6	13.0	19.2	1.7
	L_{ref}	12.9	13.6	11.1	14.2	6.9	13.6	10.1	18.2	17.3	1.7
	L_{rout}	8.7	14.6	10.3	12.3	10.8	13.9	12.2	10.5	16.6	1.2

1) calculated from the duplicates obtained for all 9 substrates in institute 2.

Table 4 - Mean values obtained by 4 institutes with the four procedures for 8 substrates (i.e. excluding vermiculite); the standard deviation of the mean $S_{\bar{x}}$ and the critical range w_4 (Dixon and Massey, 1957).

		Mean values obtained by institute					
		1	2	3	4	$S_{\bar{x}}$	w_4
Bulk dens	C_{ref}	215	226	220	216	3.9	15.6
	C_{rout}	196	228	215	218	5.6	21.9
	L_{ref}	200	197	172	171	3.8	15.0
	L_{rout}	187	216	169	166	4.6	18.1
Pore vol.	C_{ref}	85.6	87.7	88.1	85.5	0.49	1.95
	C_{rout}	86.8	87.6	88.3	85.4	0.47	1.86
	L_{ref}	86.5	89.3	90.6	88.6	0.42	1.67
	L_{rout}	87.4	88.3	90.8	88.8	0.40	1.57
water pF 1.5	C_{ref}	47.1	57.2	54.8	56.3	0.73	2.88
	C_{rout}	50.7	55.8	52.7	56.7	1.14	4.50
	L_{ref}	49.9	48.9	45.4	45.1	0.70	2.76
	L_{rout}	42.8	51.5	44.2	42.7	0.84	3.33
air pF 1.5	C_{ref}	38.4	30.5	33.3	29.2	0.88	3.48
	C_{rout}	36.2	31.8	35.6	28.6	1.34	5.28
	L_{ref}	36.6	41.4	45.2	43.5	0.92	3.63
	L_{rout}	44.6	36.8	46.6	46.1	1.10	4.36

(Values connected by a line are not significantly different)

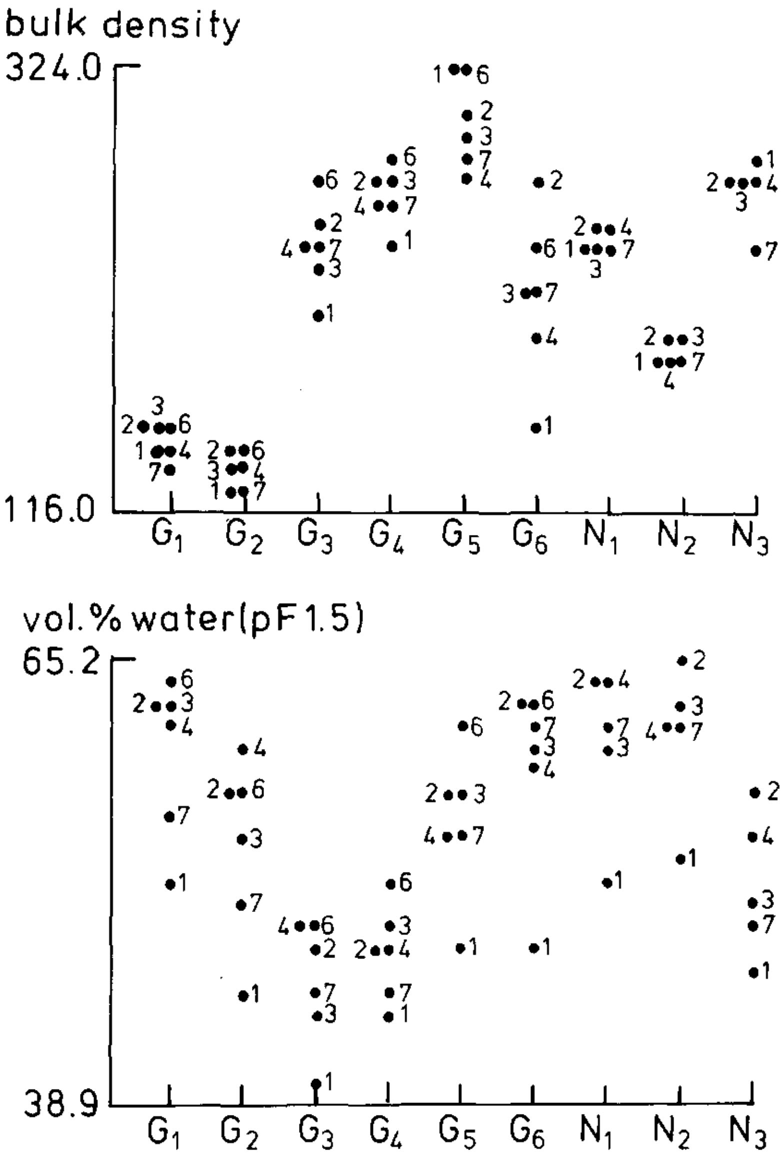

Figure 1 - Bulk density and vol. % of water at pF 1.5 found by the institutes 1, 2, 3, 4, 6, 7 for the substrates G_1 to G_6 and N_1 to N_3 when following the reference procedure C_{ref} in which compression is applied.