Soil Physical Laboratory Methods – Procedures used at the Soil Physics Laboratory 2000-2020

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Preface

This report presents the protocols and methods used 2000-2020 in the Soil Physics Laboratory at the Department of Soil and Environment, Swedish University of Agricultural Sciences, Uppsala, Sweden. The aim is to provide a transparent description of procedures used and to provide links and references to quality assurance and standards. Brief theoretical background and concepts are included for the different methods and procedures. New analytical techniques, such as integral suspension pressure (Pario) and laser diffraction (Horiba) methods for particle size distribution and pF laboratory station (Ecotech) for water retention properties, have been tested since 2020, but are not included in this report. For these, see technical manuals and scientific reporting.

Table of contents

Preface **3**

1. Particle size distribution (including loss on ignition and organic matter content) **6**

1.1 Soil sample preparation 6

1.2 Pre-sieving 9

1.3 Pipette method (sedimentation) 10

1.4 Wet-sieving 12

1.5 Weighing crucibles from pipette sampling and wet-sieving, and from dry-sieving the 0.2-0.6 mm fraction 13

1.6 Loss on ignition and organic matter content 14

1.7 Input data and calculations 15

2. Particle density **17**

2.1 Soil sample preparation 17

2.2 Measurement procedure 17

2.3 Calculations 18

2.4 Special soils (e.g. soils with high organic carbon content) 19

3. Soil water content and dry bulk density (including porosity and shrinkage) **20**

3.1 Dry bulk density 20

3.2 Porosity 20

3.3 Gravimetric soil water content 21

3.4 Volumetric soil water content (including actual field soil water content) 21

3.5 Shrinkage 22

4. Water retention properties (including wilting point) **23**

4.1 Soil sample preparation 24

4.2 Sandboxes for soil water tensions from near zero to 1 m 25

4.3 Suction plates for soil water tensions from 1 to 6 m (corresponding to applied suction settings of 0.1 to 0.6 bar on the suction device) 27

4.4 Pressure chambers for soil water tensions from 10 to 150 m (corresponding to applied pressure settings of 1 to 15 bar on the pressure device) 31

5. Saturated hydraulic conductivity **36**

5.1 Soil sample preparation 37

5.2 Assembly 37
5.3 Experimental run 40

6. Contributions and acknowledgements 46

7. References and other standards 47

8. Annexes 49
8.1 Annex 1 (see section 1.1): Centrifugation 49
8.2 Annex 2 (see section 1.1): Dispersant preparation 50
8.3 Annex 3 (see section 1.2): Procedure for faster pre-sieving 51
8.4 Annex 4 (see section 1.3): (a) Theory: calculation of sampling times and depths and (b) Former sampling times and depths for pipette method 53
8.5 Annex 5 (see section 1.7): Using the program to calculate particle size distribution and loss on ignition for fine earth particles (d_p <2 mm) 54
8.6 Annex 6 (see section 1.7): Procedure for particles with d_p >2 mm in soil sample .. 62
8.7 Annex 7 (see section 4): Special sandboxes 63
8.8 Annex 8 (see section 5.3): (a) Theory: calculation of saturated hydraulic conductivity and (b) Parameter settings 65

9. Glossary: Vocabulary English - Swedish 67

1. Particle size distribution (including loss on ignition and organic matter content)

The particle size distribution of mineral components of soil is determined by sieve and sedimentation analysis of loose soil sample. The results are presented as percentages of different particle size fractions or categories according to the Atterberg classification and adapted to ISO (2009), i.e. <0.002 mm (clay; *ler* in Swedish), 0.002-0.006 mm (fine silt; *finmjäla*), 0.006-0.020 mm (medium silt; *grovmjäla*), 0.020-0.063 mm (coarse silt; *finmo*), 0.063-0.200 mm (fine sand; *grovmo*), 0.200-0.600 mm (medium sand; *mellansand*), 0.600-2.00 mm (coarse sand; *grovsand*), 2.00-20.0 mm (gravel; *grus*), >20.0 mm (cobble; *sten*). If required, the fractions 2.00-6.00 mm (fine gravel; *fingrus*) and 6.00-20.0 mm (coarse gravel; *grovgrus*) can also be determined.

The fractions are determined in several steps. Dry-sieving is performed to separate particles with equivalent diameter (d_p) smaller than 2 mm (called `fine earth´) from those with d_p greater than 2 mm (coarse fractions including gravel and cobble) - see section 1.1. The fine earth fraction (d_p <2 mm) is then used for subsequent separation steps. A series of wet- and dry-sievings are performed before and after sedimentation, to separate the fractions with d_p 0.063-2 mm (sand, including fine, medium and coarse sub-fractions) - see sections 1.2 and 1.4. A sedimentation method is applied to separate fractions with d_p less than 0.063 mm (clay and silt, the latter including fine, medium and coarse sub-fractions) - see section 1.3. Loss on ignition can also be determined during the procedure or performed in a separate analysis, from which organic matter content can be approximated - see section 1.6. This measure of organic matter content is included in the final presentation of particle size distribution - see Table 3 in section 1.7.

1.1 Soil sample preparation

Initial air-drying, grinding and dry-sieving into fractions smaller and larger than d_p 2 mm:

Soil samples that arrive at the laboratory are first air-dried in a drying room (35 ºC) for at least one week (length of time depending on sample (loose or in cylinder), volume and water content). The samples are then broken up and milled in a soil grinder, before being passed through a sieve with a 2 mm mesh to divide the sample into fractions with d_p <2 mm (fine earth) and d_p >2 mm (coarse fractions). If the sample contains particles with d_p >2 mm, dry-sieving with a 20 mm mesh is added and the mass (in grams to two decimals) of the three soil fractions (d_p <2 mm, 2-20

mm and >20 mm) is determined to give the proportion of soil in each of these fractions (see Annex 6).

Preparation for wet-sieving and sedimentation (fractions smaller than d_p 2 mm):
A 20.00 g portion of the dry-sieved fine earth fraction (d_p <2 mm) is placed in a glass beaker and preparation steps are performed to: (i) determine whether the sample contains any carbonate, (ii) remove organic matter and (iii) disperse the sample into its constituent particles. In the first step, 45 mL of deionised water are added to the fine earth (d_p <2 mm) sample in the glass beaker to make a slurry.

Detection of carbonate content:
Then two drops of hydrochloric acid (HCl, 1 M) are added to the fine earth slurry to test if the soil contains carbonate. If effervescence is noted when the HCl comes into contact with the fine earth slurry, this indicates the presence of carbonates. If required, carbonates can be eliminated before continuing sample preparation (depending on the aim of the study), by adding HCl until the bubbling ceases.

Removing organic matter:
The next step is to add 10 mL of hydrogen peroxide (H_2O_2, 35 %) to the fine earth slurry, before boiling the solution in a heated water bath (GFL 1032 device until 2021, thereafter Hydro H 19 V LAUDA) during the day to remove organic matter. If the reaction is strong, it may be necessary to add 1-octanol (1 M) to stop the reaction so that the solution does not overflow the glass beaker. It is best to add the H_2O_2 on the day before boiling the sample, stir and leave overnight, so that the reaction can take place slowly and over a period of time before heat is applied.
During boiling, the solution should be stirred to prevent soil adhering to the bottom of the beaker and burning. It may also be necessary to add more H_2O_2 if the soil contains much organic matter. The colour of the solution will indicate the presence of residual organic matter, as will any bubbles on the top. A brownish or greenish colour means that there is still organic matter present, while bubbles on the surface mean that there is still H_2O_2 to be consumed.
By the end of the day, or before proceeding with the analyses, the solution must be clear (no organic matter and no H_2O_2). If the solution has no colour but is not entirely transparent (Figure 1), this means that there is still some H_2O_2 present, with the risk that the solution will become effervescent (start bubbling) during shaking overnight (which is the next step). To avoid this, the sample should be boiled as long as necessary to give as little residual H_2O_2 as possible. In some cases, it may be necessary to boil samples for two or even three days.

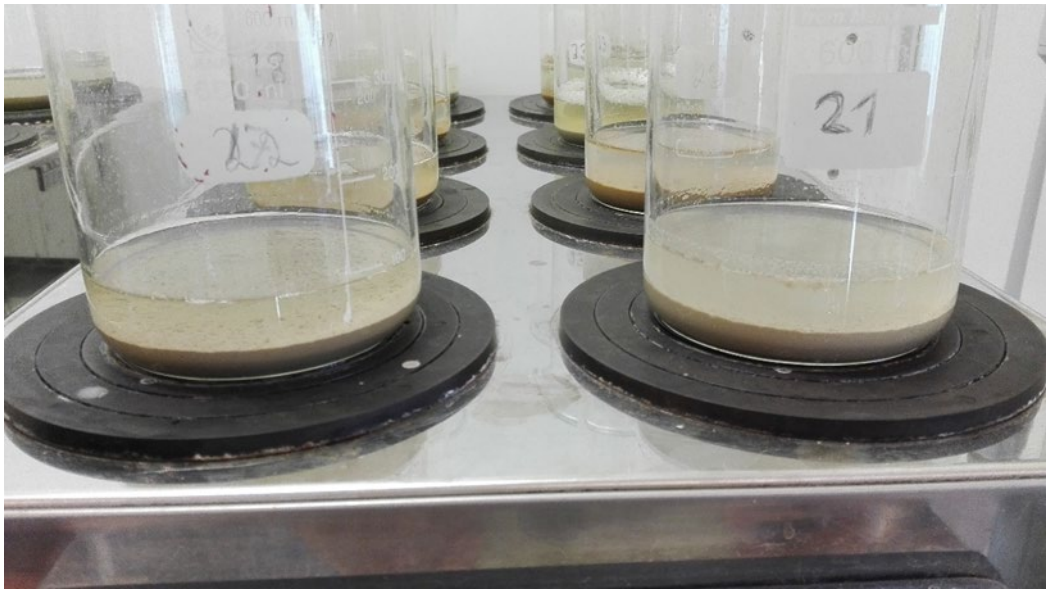


Figure 1. (Left) Sample of transparent solution ready for analysis and (right) sample with non-transparent solution requiring further boiling to remove organic matter.

If the solution still has brownish/greenish colour but is clear after the sample preparation time, the supernatant should be removed with a pipette. If the water is brownish/greenish and turbid (Figure 2), a centrifuge must be used. Centrifuging the sample will concentrate the soil particles on the bottom and the liquid part (supernatant) can be discarded. See description of the centrifugation procedure in Annex 1.

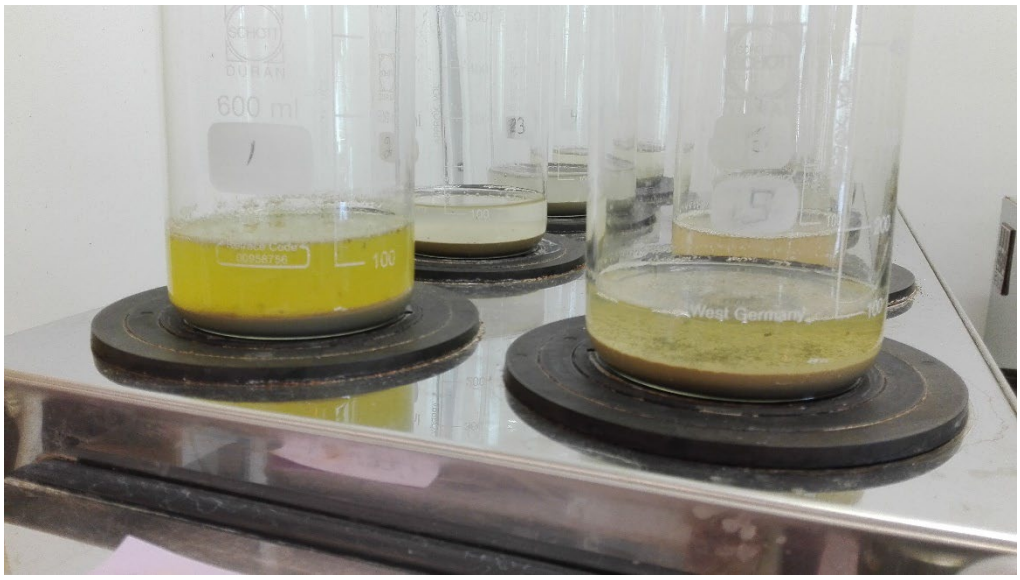


Figure 2. (Left) Sample with turbid solution that needs to be centrifuged and (right) sample with clear supernatant that can be removed with a pipette before dispersing and shaking.

Dispersing and shaking:
When the boiled solution is ready (i.e. no organic matter and no H_2O_2), it is transferred to a large plastic cylinder for shaking, together with an added dispersant (see Annex 2), in a rotator overnight.
First the glass beakers used in sample preparation are removed from the water bath and left on the bench to cool down. Once the solution is cooled to room temperature, 25 mL of dispersant are added to the glass beaker (for details of dispersant preparation, see Annex 2) and then all the sample solution is transferred to the large (1 litre) plastic cylinder. Each cylinder is sealed with a black lid and placed in the rotator (Figure 3). Care must be taken to ensure that the cylinders are properly closed, so that there is no leakage during shaking. When all the cylinders are assembled and in position, the main lid of the rotator is closed and the shaking time is set. If the samples are to be analysed on the following day, they can be shaken overnight with no pause.

Figure 3. Samples in the rotator ready for shaking overnight.

1.2 Pre-sieving

After shaking, the prepared and dispersed fine earth (d_p <2 mm) samples in the plastic cylinders need two last steps before they are ready for sedimentation measurements (pipette method, section 1.3): they are wet-sieved (0.200 mm mesh) and their temperature is brought to around 20 °C.

A support to hold the 0.200 mm aperture sieve, a plastic beaker, a plate and a brush are used for pre-sieving and a tray of metal crucibles is used to hold the medium and coarse sand samples (d_p 0.2-2 mm) for later wet-sieving. The contents of the large plastic cylinder are transferred to a plastic beaker, the large cylinder is placed

under the sieve (Figure 4) and the sample solution in the plastic beaker is poured through the sieve into the cylinder. The sieve is then angled and washed to remove sand particles retained on the mesh (fractions d_p 0.2-2 mm) into metal crucibles, for analysis by a wet-sieving procedure (section 1.4) as explained below, after the pipette method (section 1.3).
Finally, the large plastic cylinder, which now contains the fraction smaller than d_p 0.2 mm, is filled with deionised water to the 1 litre mark and placed on a round table under the pipette device (Figure 5) and left overnight. By the following the day, the samples will have reached room temperature (around 20 °C) and are ready for analysis by the pipette method.
A method for speeding up the temperature normalisation step and for determining whether pre-sieving is needed is given in Annex 3.

Figure 4. Pre-sieving at 0.200 mm.

1.3 Pipette method (sedimentation)

Once the samples are ready (i.e. large cylinder samples at 20 ºC, containing fractions smaller than d_p 0.2 mm, with water filled up to the 1 litre mark) and placed in order on the round table (Figure 5; the table can be manually rotated to sequentially position each large cylinder directly under the pipette device), the other

material that will be used in analysis is prepared. If there are 24 samples, then four trays, each with 25 crucibles (i.e. 24 plus one extra for the tare), will be needed, plus the pipette device, deionised water to replenish the pipette water reservoir, a rod (with a circular disc at the base) to stir the sample (vertical movement), a timer and a pump for the pipette device. The pipette can become dry if it is not used for some time, so it is best to pipette in some water to check that it works well and then fill the upper water reservoir with deionised water, before starting the experimental run.
When the material is ready, the pipette pump is turned on and the analysis begins. Sampling using the pipette is performed at four pre-determined time and depth steps, derived from Stokes' law for sediment velocity of different particle sizes in solution (see Annex 4a). Until 2000, the time increments used were slightly different from those used today (see Annex 4b). Around year 2000, a change was made to follow the ISO standard (particle density= 2.65 g cm^{-3}) instead of the earlier standard (particle density= 2.6 g cm^{-3}).

There are four sampling times at three different depths in the large cylinder sample. At each specific sampling time, some (larger) fraction sizes have sunk below the sampling depth, leaving smaller fraction sizes to be sampled with the pipette. At subsequent sampling times, thus, smaller and smaller fraction sizes are sampled. The following sampling times and depths are currently used (corresponding fraction sizes in brackets):
Sampling 1: 56 s at 20 cm (corresponding to the fraction with d_p <0.063 mm).
Sampling 2: 4 min 38 s at 10 cm (corresponding to the fraction with d_p <0.020 mm).
Sampling 3: 51 min 29 s at 10 cm (corresponding to the fraction with d_p <0.006 mm).
Sampling 4: 5 hours 48 min at 7.5 cm (corresponding to the fraction with d_p <0.002 mm).

Up to 24 large cylinder samples can be run simultaneously. First, for Sampling 1, each sample solution in the cylinders is stirred with the rod 15 times and the time count is started on removing the rod. The time between Sampling 1 and Sampling 2 is very short, so it is necessary to stir the samples again and restart the stopwatch before Sampling 2. For Sampling 3 and Sampling 4 this is not necessary, so the time count for those is from the start of Sampling 2.

Note: If there is only one operator, small modifications are needed: For Sampling 1, stir and start the stopwatch for each cylinder sample. For Sampling 2, stir the cylinder samples, in turn, every 2 min (in this case a maximum of 22 large cylinders can be run simultaneously, since otherwise the beginning of Sampling 3 overlaps with the end of Sampling 2). If there are two operators, one operator stirs the samples in Sampling 1 and Sampling 2, in turn, every minute and the other operator performs sampling.

At the time for each of the four samplings (depth increments) outlined above, the pipette is immersed in the sample with its valve closed. The valve is opened 2 seconds before sampling and the valve hole is blocked with a finger 1 second before sampling. Sample is drawn until the pipette is filled above the 10 mL level (excess

liquid above 10 mL level is drained back to the large cylinder through a parallel pipe), the valve is closed and the pipette is withdrawn. The sample in the pipette is transferred to a crucible by opening the valve. Some water from the upper water reservoir (uppermost on the pipette device in Figure 5) is then poured through to rinse the pipette, and then the crucible is returned to the tray.

After completing each sampling round (Samplings 1-4), the trays of crucibles are placed in the oven at 105 ºC and left to dry overnight. Each crucible is weighed on the following day (see section 1.5).

Figure 5. Samples on the table ready to start the pipette method experimental run.

1.4 Wet-sieving

When the last sample has been taken in the pipette method, the next step is wet-sieving. The following equipment is needed: sieves with 0.200 mm and 0.063 mm mesh (Figure 6), a funnel that is placed under the sieves and connected to the outlet pipe (which collects fractions not retained in the sieves), a ceramic bowl to collect the particles that are washed off the sieves, two trays of clean metal crucibles and the tray of crucibles containing the medium and coarse sand fractions (d_p 0.200-2 mm) obtained in pre-sieving (see section 1.2) before the pipette method. A water pistol connected to the tap with deionised water is also needed.

The 0.200 mm sieve is placed above the 0.063 mm sieve, which in turn is placed above the funnel (Figure 6). All the d_p <0.2 mm fraction sample solution from the 1 litre cylinder and also the d_p 0.2-2 mm sand fraction obtained in pre-sieving are poured onto the upper 0.200 mm sieve. The excess solution with particles d_p <0.063 mm that passes through the lower sieve is drained away via the funnel to the outlet pipe.

The water pistol is used to move any sand retained on the 0.200 mm sieve (d_p 0.2-2 mm) from one side of the sieve to the other and back, to gather the sand on one side of the sieve. This sand is transferred to the ceramic bowl, and from the bowl to an empty crucible taken from the first tray of crucibles. The water pistol is used again to move the fine sand particles on the 0.063 mm sieve (d_p 0.063-0.2 mm) to one side of that sieve and back, from where they are transferred via the clean ceramic bowl to a fresh crucible, taken from the second tray of crucibles.
Both sieves are then cleaned in an ultrasonic bath.

The two trays of crucibles are placed in the oven at 105 ºC and left to dry overnight. Each crucible is weighed on the following day (see section 1.5).

Figure 6. Sieves on top of the funnel for wet-sieving.

1.5 Weighing crucibles from pipette sampling and wet-sieving, and from dry-sieving the 0.2-0.6 mm fraction

On the following day, the four trays of crucibles from the pipette method (d_p <0.002, <0.006, <0.020, <0.063 mm) are removed from the oven in which they were dried overnight and placed in a desiccator chamber until they reach room temperature. Each crucible is then weighed using a laboratory balance measuring to four digits (see example of input protocol for mass of particle size fractions in Table 1 in section 1.7).

Note: All crucibles on each tray can be tared against a special tare crucible on each specific tray. Usually it is the last crucible on the tray (lower right corner) which has a dot next to the number on the bottom.

Next, the two trays from wet-sieving (d_p 0.063-0.2, 0.2-2 mm) are placed in the desiccator chamber until they reach room temperature and then each crucible is weighed using a laboratory balance measuring to two digits. After weighing, each

of the crucibles containing the d_p 0.2-2 mm sand are emptied into a 0.600 mm sieve for dry-sieving. The d_p 0.2-0.6 mm fraction that passes through the sieve is collected in a container and weighed. The mass of the fraction with d_p 0.6-2 mm is calculated by the program (see Annex 5) as the difference in mass between the d_p 0.2-2 mm fraction and the d_p 0.2-0.6 mm fraction (see also Tables 1 and 3 in section 1.7).

Maintenance of crucibles (including crucibles in section 1.6):
The metal crucibles used for holding samples need to be weighed at least twice per year, depending on frequency of use. Due to coatings and dishwasher wear on the paint, the tare mass of the crucibles needs to be checked, and corrected if necessary. Before checking the tare mass, the crucibles are placed in 1 % HCl solution for five minutes, rinsed thoroughly three times in deionised water and allowed to dry.

1.6 Loss on ignition and organic matter content

When preparing the 20 g sample of fine earth (d_p <2 mm) for the sieving and pipette analyses (see section 1.1), separate samples are prepared for analysis of loss on ignition. For this, trays with specific metal crucibles for ignition analysis are used, with approximately 11 g of prepared air-dried fine earth (d_p <2 mm) from each sample placed in a separate crucible (the exact mass of which is then measured to two decimals). The crucibles are dried at 105 ºC overnight, transferred to a desiccator chamber and weighed (two decimals) when they have reached room temperature, with the values obtained used to calculate the water content (equation 1), which helps assess whether each sample has been sufficiently air-dried. Finally, the crucibles are placed in a furnace for ignition at 550 ºC for four hours. The crucibles are then transferred to a desiccator chamber and weighed (two decimals) when they have reached room temperature, with the values obtained used to calculate loss on ignition (equation 2).

The mass calculations are as follows (see example of protocol for calculations in Table 2 in section 1.7):

$$\textit{Water content (\%)} = (\textit{(Air-dried soil - Dry soil 105ºC) / Dry soil 105ºC)} * 100 \quad (1)$$

$$\textit{Loss on ignition (\%)} = (\textit{(Dry soil 105ºC - Ignited soil 550ºC) / Dry soil 105ºC)} * 100 \quad (2)$$

The organic matter content of mineral soil samples can be roughly calculated from the loss on ignition by subtracting a correction factor that increases in magnitude with the clay content (due to increased proportion of crystal water). The relationship between organic matter (*OM*), loss on ignition (*Loi*) and clay content (*Clay*) used in the software program of our laboratory is (Ljung, 1987):

$$\textit{If Clay is } \leq 20\%: \quad OM = Loi - (0.1 * Clay) \quad (3)$$

$$\textit{If Clay is } > 20\ \%: \quad OM = Loi - (1.06 + (0.047 * Clay)) \quad (4)$$

These relationships are based on the following average correction factors (c_f) as related to clay content (%): *Clay* <15% c_f = 1.0; *Clay* 15-25% c_f = 2.0; *Clay* 25-40% c_f = 2.5, *Clay* 40-60% c_f = 3.5, *Clay* >60% c_f = 4.5.

1.7 Input data and calculations

For each soil sample analysed, all masses, i.e. the weighed soil in crucibles (tare mass deducted) for each particle size fraction (derived from pipette method sedimentation, dry- and wet-sieveing) and for loss on ignition are entered into separates rows in dedicated protocols (see examples in Table 1 and Table 2).

*Table 1. Example of an input protocol for mass of fine earth (d_p <2 mm) particle size fractions and of dry and ignited soil (*provbeteckning = *sample ID;* glödförlust = *loss on ignition;* tara = *tare mass;* invägt = *mass of dry soil at 105 °C;* utvägt = *mass of ignited soil at 550 °C)*

	A	B	C	D	E	F	G	H	I	J	K	L	M
1		**MEKANISK ANALYS (pipettmetoden)**											
2													
3				g				g			glödförlust - tara		
4	prov	Provbeteckning	Nivå								invägt	utvägt	
5	nr			d<0,002	d<0,006	d<0,02	d<0,06	0,06-0,2	0,2-0,6	0,2-2,0	g	g	
6	1	Soil A		0.0977	0.125	0.1497	0.1683	0.68	1.23	1.81	10.03	9.28	
7	2	Soil B		0.1116	0.139	0.1631	0.1832	0.51	0.96	1.42	10.77	10.17	
8	3	Soil C		0.0578	0.0714	0.0927	0.1167	4.09	2.77	4.08	10.39	9.98	
9	4	Soil D		0.0649	0.0792	0.0998	0.1239	3.85	2.62	3.68	11.3	10.89	
10	5	Soil E		0.0442	0.0553	0.0736	0.1	4.79	3.45	4.73	10.77	10.38	
11	6	Soil F		0.0448	0.0568	0.0756	0.1031	4.73	3.38	4.72	10.67	10.31	
12	7	Soil G		0.0723	0.0891	0.1092	0.1341	3.58	2.09	3.09	10.21	9.9	
13													

*Table 2. Example of a protocol for calculating the water content of air-dried soil and loss on ignition, and results from dry-sieving of d_p >2 mm fractions (*provbeteckning = *sample ID;* glödförlust (glödgningsförlust) = *loss on ignition;* tara = *tare mass;* degel = *crucible;* före tork = *before drying;* 105 °C = *mass of dry soil at 105 °C;* 550 °C = *mass of ignited soil at 550 °C;* vattenhalt = *water content;* sållning (ej tvättat) = *dry-sieving)*

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
1		**MEKANISK ANALYS (glödgningsförlust)**													
2															
3			glödförlust				glödförlust - tara			Vattenhalt	Glödförlust	Sållning (ej tvättat)			
4		Provbeteckning	tara degel	före tork	105°	550°	före tork	105°	550°			<2	2 - 20	>20	
5			g	g	g	g	g	g	g	%	%	g	g	g	
6	1	Soil A	36.92	47.22	46.95	46.2	10.3	10.03	9.28	2.7	7.5	1097.73	3.7	0	
7	2	Soil B	38.43	49.5	49.2	48.6	11.07	10.77	10.17	2.8	5.6	1260.09	4.5	0	
8	3	Soil C	36.96	47.54	47.35	46.94	10.58	10.39	9.98	1.8	3.9	1936.2	86.34	0	
9	4	Soil D	38.38	49.92	49.68	49.27	11.54	11.3	10.89	2.1	3.6	1730.16	65.42	31.26	
10	5	Soil E	36.94	47.86	47.71	47.32	10.92	10.77	10.38	1.4	3.6	1381.85	102.52	16.2	
11	6	Soil F	38.44	49.27	49.11	48.75	10.83	10.67	10.31	1.5	3.4	1244.29	106.49	48.26	
12	7	Soil G	36.95	47.41	47.16	46.85	10.46	10.21	9.9	2.4	3.0	1210.71	35.99	21.99	
13															

The program currently used in our laboratory for soil particle size distribution analysis is called Hyspec2.Exe. See Annex 5 and 6 for how to use the program.

An example of the results (output) for fine earth samples (d_p <2 mm) produced by the soil particle size distribution analysis program is shown in Table 3. When there are particles >2 mm present in the sample, the proportions of fine earth (d_p <2 mm), gravel (d_p 2-20 mm) and cobbles (>20 mm) can be re-calculated as shown in Annex 6.

*Table 3. Output protocol for particle size distribution of a fine earth (d_p <2 mm) sample, loss on ignition and organic matter content, all in % (*ler = *clay;* finmjäla = *fine silt;* grovmjäla = *medium silt;* finmo = *coarse silt;* grovmo = *fine sand;* mellansand = *medium sand;* grovsand = *coarse sand;* glödgn. förlust = *loss on ignition;* fel = *error;* mullhalt = *organic matter content;* kumulativ procent = *cumulative percentage)*

	A	B	C	D	E	F	G	H	I	J	K	L
1	**Mekanisk analys (fraktionerna angivna i %)**											
2												
3	Finjord <2mm	Ler	Fin mjäla	Grov-mjäla	Fin-mo	Grov-mo	Mellan-sand	Grov-sand	Glödgn.	Fel%	Mull-halt	
4			0,002-	0,006-	0,02-	0,06-	0,2-	0,6-	förlust			
5		d<0,002	0,006	0,02	0,06	0,2	0,6	2	%			
6	Soil A	48.1	14.9	13.6	10.2	3.6	6.5	3.1	7.5	1.5	4.3	
7	Kumulativ procent	48.1	63	76.6	86.8	90.4	96.9	100				
8												
9	Soil B	52.9	14.3	12.5	10.5	2.6	4.9	2.3	5.6	4.5	2.1	
10	Kumulativ procent	52.9	67.2	79.8	90.2	92.8	97.7	100				
11												
12	Soil C	25.7	7.3	11.5	12.9	21.3	14.4	6.8	3.9	-0.2	1.7	
13	Kumulativ procent	25.7	33	44.5	57.4	78.7	93.2	100				
14												
15	Soil D	29.4	7.7	11	12.9	20	13.6	5.5	3.6	0	1.2	
16	Kumulativ procent	29.4	37.1	48.1	60.9	80.9	94.5	100				
17												
18	Soil E	18.8	6.1	10	14.5	25.5	18.4	6.8	3.6	-2.4	1.9	
19	Kumulativ procent	18.8	24.9	34.9	49.4	74.8	93.2	100				
20												
21	Soil F	18.8	6.5	10.2	14.9	24.8	17.7	7	3.4	-1.4	1.6	
22	Kumulativ procent	18.8	25.4	35.5	50.4	75.2	93	100				
23												
24	Soil G	33	8.9	10.6	13.2	18.4	10.7	5.1	3	0.3	0.5	
25	Kumulativ procent	33	41.9	52.6	65.7	84.1	94.9	100				
26												

2. Particle density

Determining the particle density of a soil sample requires the mass and volume of the solid particles in a sample to be carefully measured. Mass is determined by weighing the soil sample. Volume is determined by the liquid displacement method, i.e. measuring the volume of ethanol (D-sprit 95%) needed to fill a flask with specific volume 50 mL containing a certain mass of soil sample. The ethanol fills all pore spaces within the soil particles in the sample, and the volume of solid soil is thus calculated as the difference between the flask volume (50 mL) and the volume of added ethanol.

2.1 Soil sample preparation

Fine earth samples (approximately 65 g) for particle density determination are prepared by air-drying, grinding, sieving with 2 mm mesh (see section 1.1) and oven-drying at 105 ºC overnight.

2.2 Measurement procedure

When the soil sample (prepared fine earth d_p <2 mm) has been oven-dried, two flasks (replicates) are prepared for each sample. A laboratory balance is used to add 30.00 g of soil to each flask. If the soil is very light or if there is not enough soil sample, less than 30 g of soil may be added per flask. *The exact mass of soil used is always carefully recorded*.

A magnet (of known volume) is added to the flask (for stirring the sample) and 35 mL of the ethanol are added using a digital burette (bottle-top Titrette 50 mL class A precision) and the exact volume (two decimals) of added ethanol is recorded (Figure 7). Each flask is sealed with an airtight cap and stirred on a magnetic plate for approximately 30 seconds (Figure 8) to get rid of air bubbles in the soil. All flasks are then placed in a controlled-temperature water bath (Grant Y28 bath until 2021, thereafter Grant thermostatic bath T100, ST18 tank and C1G immersion cooler) and left overnight for the temperature to stabilise at 20 ºC, and to allow absorption of the ethanol into the finest pores. Samples with a high organic matter content are left to react for a longer period (e.g. over a weekend). On the following morning (or after a longer period if needed), the flasks are stirred again for approximately 30 seconds, until the last air bubbles are gone, and placed back in the controlled-temperature water bath. In the afternooon, when the supernatant is

clear again, ethanol is added to the flask to bring the level to the line indicating 50 mL volume. Again, the exact volume of ethanol added (two decimals) is recorded.

Figure 7. Add ethanol.


Figure 8. Stir the sample.

2.3 Calculations

The following equations are used:

$$Total\ flask\ volume = Soil\ sample\ particle\ volume + Magnet\ volume + Alcohol\ volume \quad (5)$$

where *flask volume* = 50 mL, *magnet volume* = 0.83 mL and *total flask volume* – *magnet volume* = 49.17 mL.

The particle density (ρ_s) (*mass of dry soil* / *volume of solid soil*) is calculated as:

$$\frac{Mass\ of\ dry\ soil}{Volume\ of\ solid\ soil} = \frac{g\ of\ dry\ soil}{Total\ flask\ volume - Magnet\ volume - Alcohol\ volume} = \frac{g\ of\ dry\ soil}{49.17\ mL - Alcohol\ volume} \quad (6)$$

where *dry soil* refers to oven-dried (at 105 °C) soil.

Example:

Sample	Flask	*Dry soil* (g)	*Added alcohol 1* (mL)	*Added alcohol 2* (mL)	*Total alcohol* (mL)	*Particle density* (g mL^{-1}= g cm^{-3})
A	1	30.00	35.09	2.03	37.12	2.49[1]
	2	30.00	35.14	1.99	37.13	2.49[2]

[1]*Particle density* = 30 / (49.17 – 37.12) = 2.49 g cm^{-3}.
[2]*Particle density* = 30 / (49.17 – 37.13) = 2.49 g cm^{-3}.

2.4 Special soils (e.g. soils with high organic carbon content)

In particle density determination for some particular (special) soil types, sample preparation may differ. For example, soil samples known to have a high organic matter content are not oven-dried, only air-dried, and instead a subsample is used to calculate the water content (i.e. air-dried minus oven-dried sample mass) in order to correct the volume in the calculations. The equation used to correct the particle density $\rho_{s,w}$ considering the water content is:

$$\rho_{s,w} = (b * d) / (c * (e - f - (0.85 * b) + (0.85 * (b * d / c)) - a)) \quad (7)$$

where *a* is volume of alcohol used, *b* is mass of air-dried soil in the flask, *c* is mass of air-dried soil in the sample used for water content determination, *d* is mass of oven-dried soil in the sample used for water content determination, *e* is flask volume, i.e. 50 mL, and *f* is magnet volume, i.e. 0.83 mL.

Another example of a special soil is peat soil with large hard charcoal-like concretions that cannot be crushed by the ordinary grinder and need to be broken down to a powder using a special grinder (old mill Janke & Kunkel KG, IKA WERK; new mill IKA A10 basic) (Figure 9).

Figure 9. Specialist mill used for crushing peat soil samples containing hard charcoal-like concretions.

3. Soil water content and dry bulk density (including porosity and shrinkage)

The parameters described in this section are all related to the analyses of soil water retention in section 4, but can each be measured as independent soil properties. For preparation of cylinder soil core samples, see section 4.1.

3.1 Dry bulk density

Dry bulk density (ρ_d) is determined on undisturbed cylinder soil samples by oven-drying the samples at 105 ºC for three days. Calculations are based on the mass (in grams, to two decimals) after drying (m_s) (reduced with any cylinder tare mass) and the cylinder or soil core volume (V):

$$\rho_d = m_s / V \quad (8)$$

where ρ_d can be expressed as e.g. g cm^{-3}, kg dm^{-3} or kg m^{-3} (depending on the units used in equation 8).

Note: After the drying procedures above, the samples are taken out of the oven and placed in a grey box covered with a metal plate to prevent humidity in the air entering the dried samples. When the samples have cooled down, they are weighed.

3.2 Porosity

Porosity (Φ) is the ratio between volume of pores (V_n) and total soil volume (V):

$$\Phi = V_n / V \quad (9)$$

where Φ is dimensionless, but can be expressed in percentage by volume if multiplied by 100.

It is relatively difficult to measure V_n experimentally, and therefore to determine porosity using equation 9. However, it can be calculated from the dry bulk density (ρ_d) (see equation 8) and particle density (ρ_s) (see section 2), as:

$$\Phi = 1 - (\rho_d / \rho_s) \quad (10)$$

where Φ is dimensionless, but can be expressed in percentage by volume if multiplied by 100.

3.3 Gravimetric soil water content

Original loose, augered or cylinder soil sample is weighed (mass in grams, to two decimals), oven-dried overnight (small loose samples) or for three days (cylinder samples) at 105 ºC, and then weighed again.

Note: According to Topp and Ferré (2002), for a few cm of loose sample 24-48 h is an adequate drying time in a forced air circulating oven.

The soil water content based on mass wetness (w) is then calculated from mass before (m) and after (m_s) oven-drying (deducting any tare mass):

$$w = (m - m_s) / m_s \quad (11)$$

where w is dimensionless, but can be expressed in percentage by mass if multiplied by 100.

3.4 Volumetric soil water content (including actual field soil water content)

Volumetric soil water content based on volume wetness (Θ) is the soil water content estimated e.g. in soil water retention analyses on cylinder samples (see section 4). It is determined on undisturbed cylinder soil samples by oven-drying the samples at 105 ºC for three days. Calculations are based on the mass (in grams, to two decimals) before drying (m), the mass after drying (m_s) (deducting any tare mass), the density of water (ρ_w) (approximated to 1 g cm^{-3}) and the cylinder or soil core volume (V):

$$\Theta = ((m - m_s) / \rho_w) / V \quad (12)$$

where Θ is dimensionless, but can be expressed in percentage by volume if multiplied by 100.

A special case is estimation of actual soil water content at field sampling occasion on its volume wetness (Θ_f):

$$\Theta_f = ((m - m_s) / \rho_w) / V \quad (13)$$

where Θ_f is dimensionless, but can be expressed in percentage by volume if multiplied by 100.

In addition, the volumetric soil water content (Θ) (e.g. for wilting point determination, which is performed on loose soil) can be calculated from the gravimetric soil water content (w) and dry bulk density (ρ_d), both as determined above (section 3.3 and 3.1, respectively), as:

$$\Theta = w * (\rho_d / \rho_w) \quad (14)$$

where Θ is dimensionless, but can be expressed in percentage by volume if multiplied by 100.

3.5 Shrinkage

In the analyses described in sections 3.1 and 3.4, after final drying at 105 ºC the soil core sample in the cylinder may have shrunk to a volume smaller than the cylinder volume. In order to correct for this change, the actual volume of the dried soil (V_d) core can be calculated by measuring its diameter (D) and height (h) and using the relationship:

$$V_d = \pi * (D / 2)^2 * h \quad (15)$$

The shrinkage (S) can then be calculated from the actual volume of dried soil (V_d) and the cylinder volume (V) as:

$$S = (1 - (V_d / V)) * 100 \quad (16)$$

where S is in percentage by volume.

4. Water retention properties (including wilting point)

To measure water retention properties of a soil, different devices are used in our laboratory to apply different suctions or air pressures to soil samples in order to achieve different soil water tensions in the sample (expressed in meter water column (m), kPa or bar, where 1 m = 10 kPa = 0.1 bar). The soil water tension in the sample is expressed as a positive value, which corresponds to a negative soil water pressure in relation to atmospheric pressure (i.e. it is smaller than atmospheric pressure). The upper and lower thresholds set for plant-available water (moist soil) vary somewhat in the literature, but it is generally defined as being between soil water tension 1 m and 150 m. For soil water tensions below 1 m (wet soil), soil water is easily drainable and therefore difficult for plants to capture, while at soils water tensions above 150 m (dry soil) soil water is too tightly bound and plants cannot absorb it.

Boxes with sand beds (Eijkelkamp Sandbox 08.01) (Figure 10) are used to apply relatively small suctions to the soil sample from near zero to 1 m, thus giving soil water tensions from near zero to 1 m. These sandboxes apply suction to the soil samples with the aid of a U tube with an overflow device that can be moved along a vertical measuring scale, thereby creating gravitational outflow until equilibrium between the applied suction and tension in the soil sample is achieved. Two special devices with porous plates developed in our laboratory were used in the past for applying suctions from near zero to 7.5 cm (tension near zero to 7.5 cm) (see Annex 7).

Pots with ceramic porous plates are used for creating intermediate tensions in the soil samples (tensions 1 to 6 m), while pressure plate extractor chambers (Soilmoisture Equipment Corp., Cat.#1500 and Cat.#1000, and Tord Erikssons Svets & Mekaniska AB, Brunna) are used for creating the largest tensions (10 to 150 m). The pots apply suction with the aid of a pump, whereas the chambers apply positive air pressures.

When several suction/pressure steps are applied in turn to the same soil sample, the sequential order is from lower to higher suction/pressure. The procedure followed is the same for all devices at each suction/pressure step applied, i.e. water leaves the soil samples through the beds and plates and when there is no more water emerging (three days of similar values in the water collector), the soil samples are removed from the device and weighed. After measurement of all suction or pressure steps, the soil samples are oven-dried for three days and then weighed again, to

obtain a dry mass value to calculate dry bulk density (see section 3.1) and the water retention values for each suction and/or pressure step (section 3.2-3.4).

For the suction measurements (i.e. those generating soil water tensions from zero to 6 m), undisturbed cylinder soil samples are normally used, in order to reflect the structural properties affecting water retention. A special case is wilting point (tension 150 m) in the pressure chamber, for which loose prepared fine earth (air-dried, milled and sieved at 2 mm) is used, since soil structure is not relevant for the small pores that retain water at such high tension in soil. In the tension range 10-150 m, undisturbed samples are generally used for tensions 10-50 m, while loose prepared fine earth is generally used for tensions 50-150 m, but this also depends on the purpose of each particular study.

4.1 Soil sample preparation

When samples for water retention measurements arrive in soil cylinders, they need to be prepared before being placed for runs in the sandboxes, pots and/or chambers. First, any excess soil adhering to the outside of the cylinder is removed with a wet cloth. The soil sample surfaces are then trimmed (e.g. with a knife) to remove excess soil and level the surfaces, with due care taken to minimise soil surface smearing. If there is a depression or gap in the soil sample surface, this is measured with a ruler so that the exact volume of the sample can be estimated. The same procedure is applied if the soil surface bulges over the cylinder edge and cannot be trimmed off.

When the sample has been prepared, it is weighed for determination of actual field soil water content, if required (see section 3.4). Then a filter paper and a cloth are placed on the bottom surface, with a rubber band to hold them in place, and a yellow plastic lid is fitted on the top. The cylinder is then placed in a plastic box with the other cylinder samples for saturation (with the yellow lid loosely tilted on top to release air or any earthworms in the sample). The saturation time depends on the type of soil. Clay soils may need around two weeks (1-2 weeks for clayey soils according to Eijkelkamp (2007, 2022) sandbox instructions), whereas sandy soils saturate faster (2-3 days according to Eijkelkamp (2007, 2022)). For all types of soil, water is added step-by-step to the box, starting with 1 cm of water in the bottom of the box (saturation from the bottom upwards for all cylinder samples), to enable air to be slowly pressed out through the top of the samples. Normal tap water that has been boiled and cooled to minimise the content of air bubbles is used for saturation.

4.2 Sandboxes for soil water tensions from near zero to 1 m

The sandbox is used to apply a range of suctions from near zero to 1 m (Figure 10). It is recommended to read the instructions in the manual provided by the company (Eijkelkamp, 2007, 2022) before use. The instructions provided below are a summary including adaptations to the routines in our laboratory.

Figure 10. Sandbox (model 08.01, Eijkelkamp) used to apply relatively small suctions to soil samples (from near zero to 1 m).

Maintenance:
When the sandboxes are not in use they need to be saturated with water (i.e. water level kept above the level of the sand surface), since otherwise air enters the sand and pipes.
The cloth placed on the sand surface in the box must be washed often to remove any dirt originating from the samples, so that the meshes do not clog.
The sand in the box must also be washed occasionally. For a partial cleaning, 2 cm of sand are removed from the top layer and placed in a bucket of warm water, stirred and allowed to settle for 2-3 hours (previously they were allowed to settle overnight, but that allowed any clay and dirt present to settle). The dirty water is removed and the process is performed twice more.
For a full general cleaning, all the sand in the sandbox is removed and washed, and the cloth is replaced with a new one. In practice, full cleaning is done when the boxes are apparently malfunctioning.

Note: Two different types of sand are used in the sandboxes, brown (older) and white (newer), and the sand texture may differ slightly between these two types. Therefore when refilling is needed, sand of the same colour as the original is used.

Before experimental run:
Before applying suction in the actual experimental run, a check is made to ensure that there is no air in the system. This is done by draining the box and observing whether there are any air bubbles in the pipes.

Note: The sandboxes currently used in our laboratory are a modified version of the original in that a reading cylinder (measuring beaker) has been added to measure the amount of water emerging from the sandbox.

Experimental run:
The water level in the sandbox at the start of the experimental run should be 1 cm above the sand surface. The cylinders are placed on the cloth on the sand surface, ensuring good contact, and the sandbox is covered with the main lid.
Before opening the valve to drain (black lever in Figure 10), the suction is set by moving the suction overflow regulator to the desired level on the vertical ruler (right side of sandbox in Figure 10). This level should be counted from the middle of the cylinder. For example, to apply 100 cm suction to a 5-cm cylinder, the overflow regulator should be placed 97.5 cm below the sand surface level (= bottom of cylinder) (100-(5/2)). For a 10-cm cylinder, the overflow regulator should be placed 95 cm below the sand surface level (100-(10/2)).
Once the suction is set using the overflow regulator, the valve is opened and the sample begins to drain, with the excess water initially collected in a bucket. After a few hours, a measuring beaker is used to collect the water instead of a bucket, in order to record the amount of water draining out per day. After three days in a row with the same reading in the measuring beaker (i.e. no further drainage), the soil water tension in the samples is assumed to have equilibrated with the actual suction step and the samples are ready to be weighed.
After weighing, the samples are placed back in the sand box and the next suction step to be applied is set using the suction overflow regulator. The procedure described above is then followed for the new suction step.

Note: A modification compared with the procedure (manual) used with the original sandbox is that when the samples are removed for weighing after each suction step, the sandbox is saturated again before the next measurement at a higher suction, in order to maintain good hydraulic contact between the sample and the sand bed.

Note: Peat soil is special. The measurements may take a longer time than for mineral soils.

After completion of the runs in the sandboxes, the soil cylinder core samples are either used again for runs at higher tensions (see sections 4.3 and 4.4) and/or for determination of saturated hydraulic conductivity (section 5). If those runs are not desired, the samples are oven-dried to enable calculation of dry bulk density and volumetric water content (see section 3).

4.3 Suction plates for soil water tensions from 1 to 6 m (corresponding to applied suction settings of 0.1 to 0.6 bar on the suction device)

The pots with ceramic porous plates used for tensions of 1 to 6 m are connected to a vacuum system in our laboratory building (Figure 11). The soil cylinder samples are placed on the ceramic plate inside the pot. The plate is connected to a water outlet pipe which leads to a graduated cylinder, where the amount of water collected every day can be recorded. A white quartz meal (powder) slurry is applied to the plate before placing the cylinder samples on top, to improve the contact between samples and plate.

Simultaneous use of up to five pots, each with individually applied suction (A, B, C, D and unlabelled), is possible (Figure 12). The vacuum system operates at approximately 0.9 bar (which is equal to 9 m suction) (Figures 13 and 14). The suction power comes from a grey tap on the laboratory bench (Figure 13), which is connected to a reservoir tank (yellow-labelled pipe on the tank) (Figure 15), and to the pressure regulator (red-labelled pipe).
The yellow- and green-labelled pipes at the top of the panel (Figure 12 and 14) relate to a pump that is no longer in use (Figure 16).

Figure 11. Pots with ceramic porous plates connected to the laboratory vacuum system.

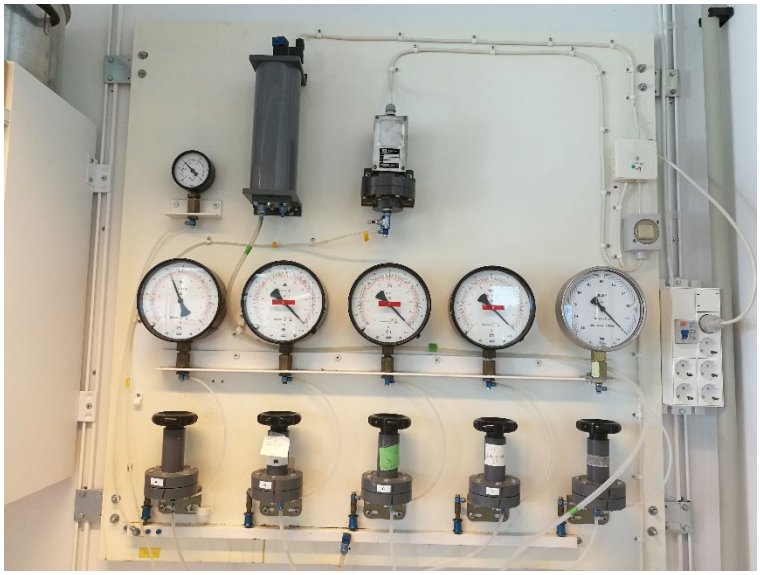


Figure 12. Valves used to adjust suction to the desired setting.

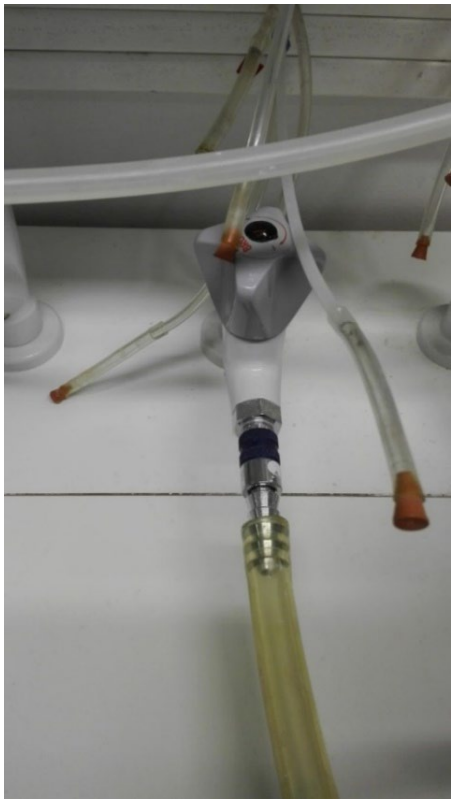


Figure 13 (left). General vacuum system.
Figure 14 (right). General pressure regulator for establishing the desired suction (on top).

Figure 15. Reservoir tank.

Figure 16. Pump formerly used in the system.

Maintenance:

The ceramic plates are kept in the pots when not in use, after thorough cleaning (no residual quartz powder or water). After use, the pots are cleaned with tap water. First, the quartz powder is removed with a rubber spatula (Figure 17), and then the pipe is disconnected and the plate is removed. The plate is washed after using it for a group of samples (no need to wash it after every single measurement). The pots are washed thoroughly occasionally and standing water in the bottom of the pot is removed, since it can damage the material. While cleaning the plate, care must be taken not to damage the edge of the plate or scratch the surface. The plate and the rubber material on the base of the plate are very tightly joined and there should be no air between them. After washing the plate, it is replaced in the pot, the small pipe is reconnected, water is poured on the plate, the lid is closed and a suction of 6 m (0.6 bar) is applied to rinse the plate.

Before experimental run:

First, the plate has to be saturated in water. Water is slowly added until the plate does not absorb more water (this step takes only a short time, 1-2 hours). Next, the white quartz powder slurry for promoting contact between the plate and the sample is prepared by mixing the powder to a semi-fluid paste with a little water, spreading it on the plate surface and adding more water to create a viscous mix that spreads over the whole plate (Figure 17).

Figure 17. Pot and (inside) plate covered with white quartz powder slurry.

Experimental run:
The samples are placed on the plate and the pot lid placed on top, to close the pot. The grey tap (Figure 13) is opened and the general vacuum pressure is checked (0.9 bar) (Figure 14). The blue valve (for establishing the suction to each regulator) is opened (Figure 12) by moving it up. To change the suction, the large black knob (pressure regulator value) is rotated (Figure 12). However, the same suctions are generally used for each pressure regulator, so they are already pre-set.
The water level is measured every day in the measuring beaker that collects the water (Figure 11). After three days in a row with the same reading in the measuring beaker, the soil water tension in the samples are assumed to be equilibrated with the actual suction step and the cylinder samples are removed and weighed.
After weighing, the samples are placed back in a pot, the next suction step is applied with the pressure regulator and the procedure is repeated.

Note: There should be no leaks in the system (pipes and connections). The small pipe connecting the vacuum system with the plate can become slightly loose over time. A small piece should be cut from the end of the pipe to fix this problem.

Note: There are different plates for different suctions. It is important to check when using a plate that the suction noted in the specifications for the plate is not exceeded (Figure 18).

Figure 18. Plate specifications.

Once the runs on the suction plates have been completed, the soil cylinder core samples are either used again for runs at higher tensions (see section 4.4) and/or for saturated hydraulic conductivity determination (section 5). If those runs are not desired, the samples are oven-dried to enable calculation of the dry bulk density and volumetric water content (see section 3).

Note: Even if possible and sometimes done, using the same undisturbed cylinder sample for both water retention (at these tensions) and saturated hydraulic conductivity measurements is not recommended, since both types of measurements may affect the pore size distribution in the sample by formation of fissures (water retention) and inner erosion (saturated hydraulic conductivity) during experimental runs.

4.4 Pressure chambers for soil water tensions from 10 to 150 m (corresponding to applied pressure settings of 1 to 15 bar on the pressure device)

Pressure plate extractor chambers are used to create the highest soil water tensions in the samples. The method used is slightly different than in previous steps, since the soil water tension in the samples is not achieved by suction, but by pressure. Furthermore, the soil samples do not have a lid on top, to allow the pressure to push the water out of the cylinder samples and through the porous plate, before finally being collected in a burette.
When applying the highest pressures (e.g. for tensions 50-150 m) undisturbed soil cylinders are not used. Instead, a small amount of loose prepared fine earth (d_p <2 mm) mixed with water is placed in a small plastic ring directly on the porous ceramic plate. In this case, no quartz powder is needed on the plate to improve the

contact between plate and sample. If undisturbed soil cylinder samples are analysed (e.g. for tensions 10-50 m), however, quartz powder is needed.

Maintenance:
When not in use, the chambers have to be kept dry and empty. The ceramic plates are stored clean and dry in a box, where they are protected from breakage or scratching. The pressure valve to each chamber must be closed, but the general pressure supply can be left open.

Before experimental run:
Before use, the plate must be saturated in water, preferably for two days, but at least overnight. For loose soil, it is also best to prepare the soil sample on the day before the experimental run. For this, 4-6 spoonfuls of prepared fine earth (d_p <2 mm) are placed in a metal cup and some water is mixed in to make a paste with the soil (Figure 19). For undisturbed soil cylinder samples (e.g. for tensions 10-50 m), the samples are prepared as described in section 4.1 above.

Experimental run:
The saturated porous plate is placed in the chamber on the supports (triangle) (Figure 20), and the drainage pipe is connected. Numbered small plastic rings are then placed on the porous plate, the loose soil paste samples are placed in rings with a spoon and finally a weight is placed on top of the soil paste (Figure 21 and 22). For undisturbed soil cylinder samples (e.g. for tensions 10-50 m), the quartz powder-water paste is prepared on the porous plate and the cylinders (with cloth, filter paper and rubber band, but without lid) are placed on top. When the sample rings, or alternatively soil undisturbed cylinders, are ready on the plate, a rubber ring is placed on the top circumference of the chamber and the chamber is closed with its main lid and sealed with the screw-nuts (Figures 23 and 24). When the chamber is ready, the red-white valve of the burette where water is collected is closed (lower smaller valve in Figure 24). The red valve to apply pressure to the chamber (upper larger valve in Figure 24) is then opened and the uppermost black valve is slowly regulated to adjust to the exact applied pressure. It takes some time for the pressure to stabilise at the desired pressure. During that time, checks are made to ensure that there are no bubbles (air leakages) in the recording burette. If bubbles are detected, the black pressure valve is closed and the chamber is opened, so that all pipe connections can be checked.

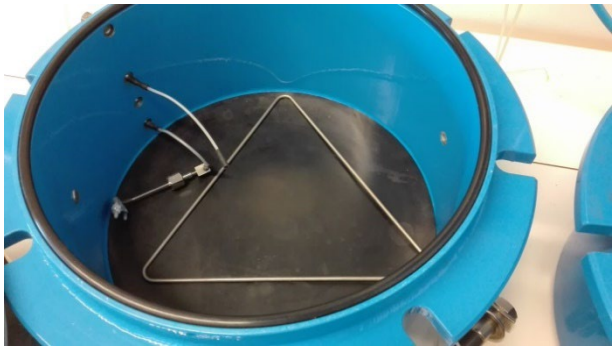
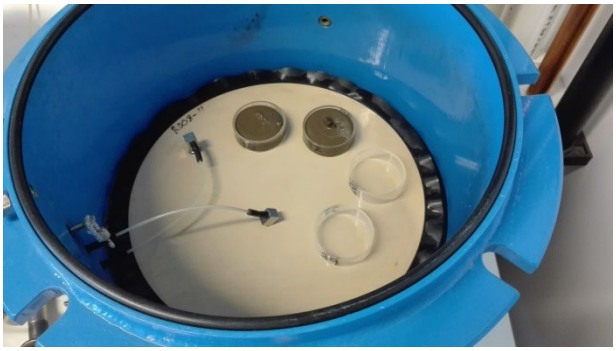


Figure 19 (left). Soil preparation for pressure plate extraction.
Figure 20 (right). Empty pressure plate chamber.



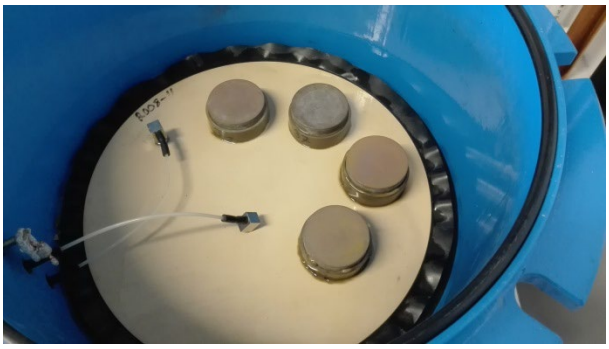


Figure 21 (left). Chamber with plate and rings with soil paste.
Figure 22 (right). Weights on top of the soil paste.

Figure 23 (left). Closed chamber.
Figure 24 (right). Sealed chamber and valves to burettes and pressure control.

Note: For small rings with loose soil, two small rings with loose soil are prepared for each sample (replicates), and the soil sample and ring number are recorded. The replicates are always run in separate chambers. In each chamber, if necessary, two plates can be placed on top of each other, separated by three small solid plastic supports.

During the run, the amount of water collected in the burette is recorded every day. When the same value is obtained three days in a row, the samples soil water tension are assumed to be in equilibrium with the actual applied pressure and the samples are removed for weighing. The measuring burette is emptied by opening the red-white valve (lower smaller valve in Figure 24) and then the black valve is gradually closed to slowly lower the pressure. When the pressure is near zero, the red valve (from the air supply) is also closed. The chamber is ready to open when no more air leaves. The soil rings (with loose soil) are removed with the help of a spatula and each soil sample is pushed carefully into a crucible (Figures 25 and 26). The soil samples are weighed (in grams, to two decimals) (Figure 27) and placed in the oven for drying at 105 ºC overnight (at least 12 hours). The soil samples are re-weighed on the following day (for determination of water content, see section 3). For undisturbed soil cylinder samples (e.g. for tensions 10-50 m), the complete procedures to estimate the dry bulk densities and water contents are described in section 3.

Figure 25 (left). Removing samples from rings.
Figure 26 (right). Tray of sample crucibles.

Figure 27. Weighing sample.

Note: The values in the recording burette (Figure 24) should increase during the process, i.e. water should keep coming out. The water level in the burette may decrease due to an occasional decrease in pressure in the chamber.

Note: For small rings with loose soil; when preparing for weighing, the plastic ring with soil and the weight on top are removed from the plate and the soil is transferred to the metal crucible used for weighing. Any soil adhering to the ring walls or to the weight should not be added, just the soil that comes together in one piece.

After the experimental run, the equipment is cleaned. The ceramic plate is disconnected, removed and washed. In the case undisturbed soil cylinder samples have been used (e.g. for tensions 10-50 m), clean the plate from quartz powder. When dry, the ceramic plate is placed back in its storage box. The valve for collecting water in the burette can be left open. The black rubber ring is removed and the main lid is replaced to keep the chamber closed.

5. Saturated hydraulic conductivity

At our laboratory, a permeameter is used to measure saturated hydraulic conductivity in soil samples. The samples used in this device are soil cores in metal cylinders, 5 or 10 cm high and with 7.2 cm inner diameter. The height level of the cylinder in the device is adjusted using a plastic ring and a plastic cross that are placed on the bottom of the metal container when assembling the samples (Figure 28).

Note: The permeameter has a system of pipes to add water to the tank. The system controls temperature and input/output water to/from the permeameter device and in the gutter sections (the latter determining the input water head level). ***Do not open or close any valves if unsure how they work****!*

During the actual experimental run, water passes through the saturated soil cylinders at a constant water head and is collected in graduated cylinders. After one hour of initial flow without measurements, the actual run starts. The time taken for approximately 80 mL to enter each graduated cylinder is recorded, and used in calculation of saturated hydraulic conductivity. If the flow is slow, the amount of water collected after 60-75 minutes is recorded. After the run, the soil cores are generally not used again for any other tests, so they are oven-dried to calculate the dry bulk density, if desired (see section 3.1).

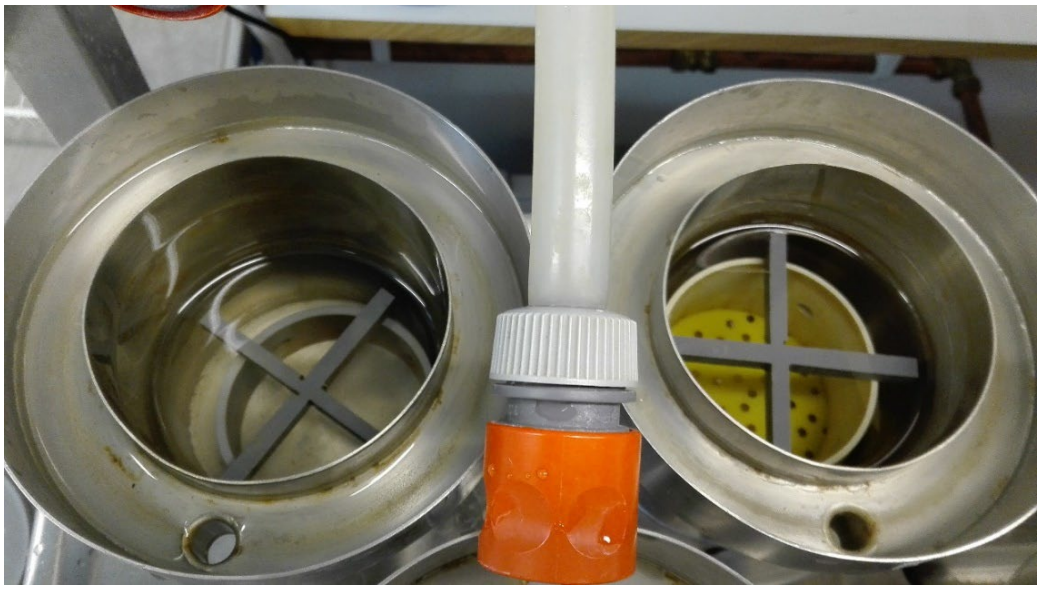


Figure 28. Container for measuring saturated hydraulic conductivity using (left) 10 cm high cylinders and (right) 5 cm high cylinders, as regulated with the height adjustment equipment at the base.

5.1 Soil sample preparation

First, the soil samples must be saturated. If the cylinder samples have been used previously for water retention measurements (section 4), they are ready to be saturated again. However, using the same sample for both water retention and saturated hydraulic conductivity measurements is not recommended (see note in the end of section 4.3). Therefore, generally, some sample preparation is required where the soil cylinders are cleaned and trimmed as described in the first paragraph of section 4.1 (note any artificial holes present that may influence the flow). Before the experimental run, a permeable tissue and a perforated (yellow) lid are attached to the bottom of each cylinder sample and a solid (yellow) lid is placed on the top (the latter is removed when the experimental run starts). The perforated lid and tissue generally enable water to pass through the sample without soil losses, but sandy soils may still suffer some losses during the experimental run in the permeameter.

When a group of samples (up to 24 at a time) are ready for saturation (see also second paragraph of section 4.1), they are placed in a grey plastic box, water is added slowly from below (i.e. 1 litre of boiled and cooled tap water every day until it reaches the upper edge of the cylinders), until the samples are saturated. This can take from a few days (sandy soils) to two weeks (clay soils). The plastic box of saturating samples is covered with a lid, but the upper (yellow) lid on each cylinder is left with a narrow open gap to allow any earthworms present to leave the sample. When the soil samples are saturated, they are ready for assembly in the apparatus for determination of saturated hydraulic conductivity.

5.2 Assembly

The saturated cylinder sample with the perforated lid and tissue attached to the bottom is removed from the plastic box and the steps in Figures 29-32 are followed (example with 5 cm high cylinder).

Figure 29. Soil cylinder sample (5 cm height) and attachments to be joined.

Figure 30. Set-up (5 cm soil samples) with black base cap (threaded sleeve to attach soil sample to the water head control black top cylinder, see Figure 29), rubber ring (seal) and upper grey ring (to concentrate water flow onto the soil sample surface).

Figure 31. Black top cylinder screwed in place onto the black base cap and tightened.

Figure 32. Samples placed back in water.

For 10 cm high cylinder samples, a shorter grey ring is used (compare Figure 33 with Figure 30).

Figure 33. Soil cylinder sample (10 cm cylinder) with parts to be joined.

When the samples are placed back in the grey plastic box (Figure 32), the water level must reach the bottom part of the black structure so that the samples saturate again overnight before the run. On the following morning, the experimental run can begin. A check should be made to ensure that the black base-top cylinder structure is tightly sealed before placing the samples in the permeameter device, i.e. tighten again the black top cylinder and the black base cap. Furthermore, the metal containers in the device (Figures 34 and 35) into which the samples are placed, must have water up to 1 cm below the upper edge, i.e. they need to be filled with water before adding the samples. When each sample is in place, it is connected to the water gutter (which controls the water head level) via the flexible pipe (Figure 34). This connecting pipe should lean a little from the gutter towards the sample, i.e. it should not be completely horizontal.

Figure 34 (left). Desired inclination of water input pipe.
Figure 35 (right). Samples in the permeameter device.

5.3 Experimental run

Once the samples are assembled in the permeameter, water is allowed to circulate for one hour before the actual measurements begin. The first step is thus to turn on the pump (Figure 36), turn on the electricity (Figure 37), open the water tap (Figure 38) and open the valves (Figure 39). A check should be made to ensure that the upper gutters are filling with water and that this water is coming through the connecting pipes to the cylinder samples (Figures 40 and 41). When all the cylinders have a constant water head, the water overflow from the gutters should not be too fast (check this in the outlet transparent pipes connecting the gutters back to the tank (Figure 42) and restrict the water flow with the valve if the water flow is too fast).

After 45 minutes water running, a check is made for leakages in the connections of the black structures above the sample. If there are leakages (Figure 43), a piece of cloth is used to collect leakage water into a separate container (Figure 44). This is necessary since if this water were to enter the metal container (i.e. not being diverted), it would be collected together with the infiltrated water and generate overestimates of the sample's saturated hydraulic conductivity. It is important to ensure that there is enough water in the metal container: when the flow rate through the sample is very slow, the water on the surface of the container may evaporate. If this happens, the water percolating through the sample will enter the metal container, but will not be collected in the graduated cylinder. To correct this, refill water is added to the metal container (Figure 45), and left for 15 minutes before restarting the experimental run.

Before starting the actual measurements, all materials needed must be ready: graduated cylinders to collect water, stopwatch, record sheet and pen to write down times and volumes, and thermometer to read the temperature of the water during the experimental run (Figure 46).

Figure 36 (left). Pump control.
Figure 37 (right). Electricity control.

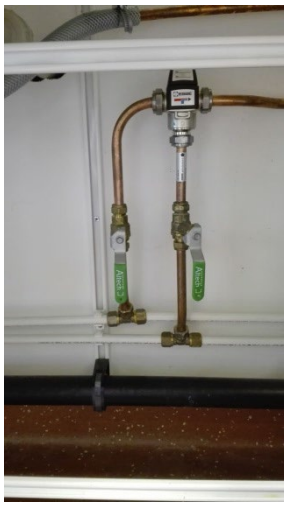


Figure 38 (left). Water tap.
Figure 39 (right). Valves.

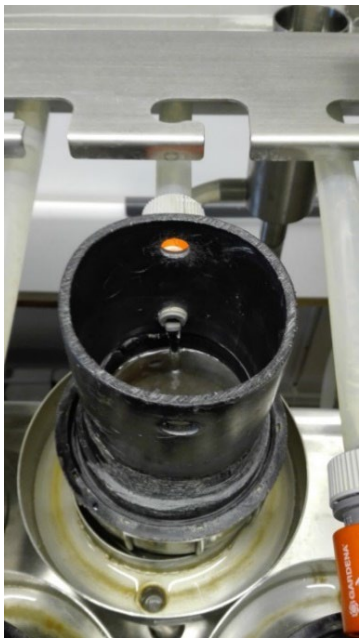


Figure 40 (left). Water filling cylinder sample.

Figure 41 (right). Water reservoir gutters (two halves) with connecting pipes.

Figure 42. Overflow (in the gutter) and outlet pipes (vertical).

Figure 43 (left). Leakage.
Figure 44 (right). Dealing with leakage.

Figure 45 (left). Refilling water in metal container.
Figure 46 (right). Recording water temperature.

To start the run, the tray with the graduated cylinders is adjusted in place so that each is centred below the outflow pipe from one soil sample set-up, and the stopwatch is started (Figure 47). The flow can be very fast (filling the graduated cylinder in less than a minute) or very slow (0 mL after one hour). If it takes less than one minute to obtain 80 mL for a sample, two further measurements are performed on that sample. If the flow rate is so high that soil in the cylinder (especially sand) moves with the water, that sample must be removed directly after the reading time, since such soil losses will affect later dry bulk density estimation. It is important to record the temperature of the water in both water gutters during the run (Figure 46).

Figure 47. Samples placed in the permeameter ready for starting the measurements.

At the end of the run, i.e. when there is 80 mL water in the graduated cylinders or after 75 minutes, the tray of graduated cylinders is pulled out from under the outflow pipes from the soil sample set-ups, the pump is stopped and the water tap is closed. For samples that did not give 80 mL, the water level in the graduated cylinders is recorded. If there are less than 10 mL, a laboratory balance is used to weigh the water and calculate the volume (after reduction for any tare mass, 1 g = 1 mL). The soil cylinder samples (including joined attachments) are removed from the permeameter device and placed in a grey plastic box with a little water. Finally, a check is made for any leakages between the rubber ring in the black structure and the cylinder, by observing if any water is being lost. If leaks are observed, all the attachments are removed and reassembled and a new experimental run is made in the permeameter.

If there is no leak, or after this extra experimental run, the measurements for saturated hydraulic conductivity are completed and the samples can go to any next step. For example, if measurement of dry bulk density is also desired, all the attachments are removed and the cylinder soil samples are placed on a metal plate

to oven-dry (see section 3.1). All the materials used (black and grey structures etc.) are cleaned for re-use.

Note: The pipes below the permeameter device conduct water to it and the valves should not be adjusted - ***keep them as in Figure 48***!

Figure 48. Permeameter valves and water tank.

For calculations of saturated hydraulic conductivity from the permeameter runs described above, see Annex 8.

Maintenance:
The permeameter does not need any special maintenance. The metal containers that hold the samples during the experimental run need to be washed now and then, because some soil can accumulate there.

6. Contributions and acknowledgements

The procedure descriptions in this manual were made by Ana María Mingot Soriano, to a large degree based on instructions received from Christina Öhman. Ingmar Messing further processed and edited the descriptions and provided additional theoretical background, concepts and calculations. Suggestions on the contents were provided by Jennie Barron and Nicholas Jarvis, and Anna Eklöv Pettersson assisted with feed-back on some of the procedure descriptions. A linguistic review, including editorial suggestions, was performed by Mary McAfee. The photo illustrations were all taken by Ana María Mingot Soriano. The work in creating this manual was funded by the Swedish University of Agricultural Sciences (SLU), partially supported by EJP SOIL (Horizon Europe Grant Agreement 862695).

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<u>Sections in Methods of Soil Analysis - Part 4, Physical Methods. 2002. Soil Science Society of America Inc., Madison, Wisconsin, USA. (Editor in-Chief: Warren A. Dick):</u>

2.1 Bulk Density and Linear Extensibility; 2.1.2 Core Method. (Grossman R.B., and Reinsch T.G.)

2.2 Particle Density. (Flint A.L., and Flint L.E.)

2.3 Porosity. (Flint L.E., and Flint A.L)

2.4 Particle-Size Analysis. (Gee G.W., and Or D.)

3.1 Water content; 3.1.3.1 Thermogravimetric Method Oven-Drying (Topp G.C., and Ferré P.A.)

3.3 Water Retention and Storage; 3.3.2 Laboratory (3.3.2.1 - 3.3.2.6). (Dane J.H., Hopmans J.W., and Romano N.)

3.4 Saturated and Field-Saturated Water Flow Parameters; 3.4.2 Laboratory Methods. (Reynolds W.D., Elrick D.E., Youngs E.G., Amoozegar A., Booltink H.W.G., and Bouma J.)

ISO-standards - Svensk standard (Swedish Standards Institute), examples:

SS-ISO 11277:2009. Markundersökningar – Bestämning av kornstorleksfördelningen i mineraldelen av jord – Sikt- och sedimentationsmetod (ISO 11277:2009, IDT). *Soil quality – Determination of particle size distribution in mineral soil material – Method by sieving and sedimentation (ISO 11277:2009, IDT)*.

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ISO-standards – International, example:

https://www.iso.org/committee/54328/x/catalogue/

8. Annexes

8.1 Annex 1 (see section 1.1): Centrifugation

Samples that still have non-clear liquid after sufficient preparation time (removing organic matter with H_2O_2) are transferred to centrifuge bottles in groups of two or four samples. The bottles (including sample) must have similar mass, since otherwise the centrifuge becomes unbalanced and does not work. The bottles are filled with sample up to two-thirds of the volume, sealed with a grey cap and placed in the centrifuge (two opposite each other, or all four). They are fixed in place with transparent caps (Figure A1) and the main lid of the centrifuge is closed.
Turn on the centrifuge power by pressing the button on the back left side. The centrifugation time and speed are set (20 minutes at 3500 rpm), a check is made to ensure that the lid of the centrifuge is closed and the "Start" button is pressed. The centrifuge speed should exceed 1000 rpm within a few seconds (Figure A2). If it slows down before that speed, some extra actions may be needed: i.e. open and close the lid again or re-organise the samples from four to two opposite at a time.
When everything works well, wait for the centrifuge to stop after 20 minutes, press the "Open lid" button and take out the bottles.
If the supernatant is clear it can be discarded. All the soil from the bottom of the bottle is then carefully transferred to a large plastic cylinder for use in the subsequent dispersion and shaking procedure. If the supernatant is not clear, all the sample (soil and supernatant) is transferred to the plastic cylinder for use in dispersion and shaking.

Figure A1. Bottles in place ready to centrifuge.

Figure A2. Centrifuge readings.

8.2 Annex 2 (see section 1.1): Dispersant preparation

The dispersant used in our laboratory is prepared in-house. It loses its properties in one month, so a limited amount is prepared each time. We currently use a mixture of sodium metaphosphate (natriumpolyfosfat), $(NaPO_3)n$, and sodium carbonate (natriumkarbonat), Na_2CO_3, as dispersant.
In the past, we used sodium diphosphate (natriumdifosfat) (0.1 M $Na_4P_2O_7 \cdot 10\ H_2O$) (see Ljung, 1987) for preparation of sodium diphosphate.

For preparation of 2 litres of dispersant ($(NaPO_3)_n + Na_2CO_3$) (Figures A3 and A4): Pour 1.4 litres of deionised water in a large glass beaker, cover and warm to 30-40 ºC (cooker setting 3), which takes around 15 minutes. Meanwhile, weigh 33 g $*$ 2 = 66 g of $(NaPO_3)_n$ and 7 g $*$ 2 = 14 g of Na_2CO_3. When the water is warm, add the 66 g of $(NaPO_3)_n$, stir with a rod, cover the beaker again and wait until the powder is dissolved, then turn off the cooker. Wait until the solution cools down and then add the 14 g of Na_2CO_3 and stir.

When the solution reaches room temperature (20 ºC), add deionised water to fill the beaker to the 2 litre mark. Finally, transfer the dispersant to a large bottle and store in a cupboard at ambient temperature (20 ºC).

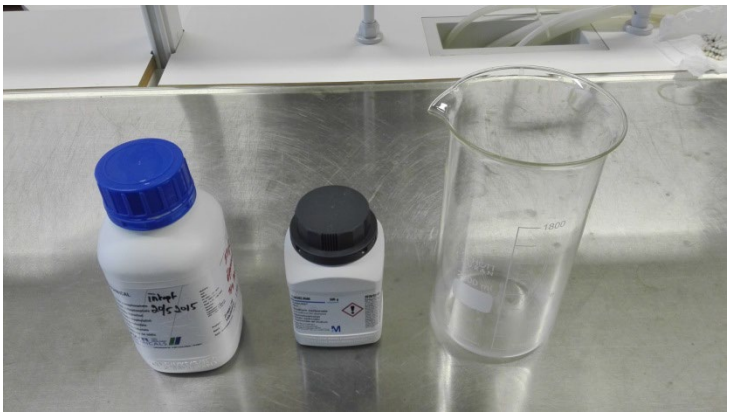


Figure A3. Chemicals used to prepare the dispersant.

Figure A4. Water warming for the dispersant.

8.3 Annex 3 (see section 1.2): Procedure for faster pre-sieving

The following is based on notes from the methodology followed by Christina Öhman until 2016 to speed up the analyses (sometimes enabling all samples to be analysed in 10 hours). However, these speeding up steps are no longer used in our laboratory, in order to follow a standardised way of analysing all samples.

Sample temperature must be 20 ºC during sedimentation. During dispersion (shaking overnight), the temperature of the samples increases. In the procedure described in section 1.2, the samples are pre-sieved on one day and then measured

on the following day, allowing them to reach room temperature. To speed up temperature normalisation so that measurements can be made on the same day as pre-sieving, the large cylinders with prepared and dispersed soil can be placed in a sink of cold water (Figure A5). If they are left too long in the sink, they may become too cold, so before placed on the round table for measuring, the temperature must be checked with a thermometer.

The following step can be taken in order to determine whether pre-sieving is needed or if it can be skipped, thereby saving time. Pre-sieving is necessary only when sand (d_p 0.2-2 mm) is present. A fast way to check for this is to examine the bottom of the large beaker after dispersion. If there is a lot of sand (Figure A6), dry-sieving is probably needed. A second check can be made when the black lid on the large cylinder is removed after dispersion. Sand on that lid is direct proof that dry-sieving is required.

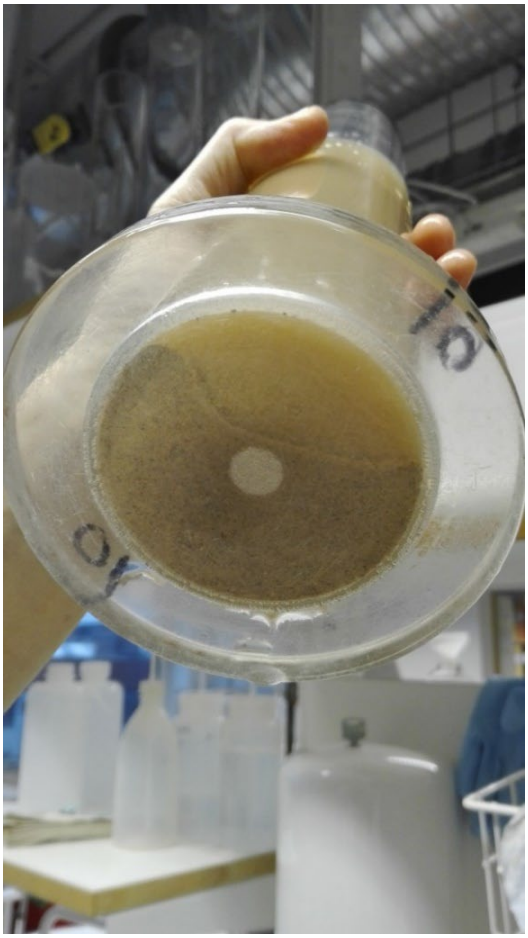


Figure A5 (left). Samples cooling down.
Figure A6 (right). Sand on the bottom of the cylinder.

8.4 Annex 4 (see section 1.3): (a) Theory: calculation of sampling times and depths and (b) Former sampling times and depths for pipette method

(a) Theory: calculation of sampling times and depths from Stokes' law

Stokes' law, adapted from Stokes (1851), describes the terminal sedimentation velocity v (m s^{-1}) of spherical particles as a function of their diameter d (m) so that:

$$v = (g * (\rho_s - \rho_f) / (18 * \eta)) * d^2 \quad \text{(A1)}$$

where g (9.81 m s^{-2}) is the acceleration due to gravity, ρ_s (kg m^{-3}) is the density of the suspended particles, ρ_f (kg m^{-3}) is the density of the liquid, and η (kg s^{-1} m^{-1}) is the dynamic viscosity of the liquid.

The equivalent diameter of particles which have reached depth z after time t, $d_p(z,t)$, where $z = v * t$, is:

$$d_p(z,t) = \sqrt{(18 * \eta) / (g * t * (\rho_p - \rho_w))} \quad \text{(A2)}$$

where ρ_p is the mean particle density (taken as 2650 kg m^{-3} (ISO, 2009)) and ρ_w is the density of the liquid containing the soil suspension (taken as 1000 kg m^{-3} (ISO, 2009)), and the corresponding time $t(d_p,z)$ is ($v = z / t$):

$$t(d_p,z) = z / ((g * (\rho_p - \rho_w) / (18 * \eta)) * d_p^2) \quad \text{(A3)}$$

For example, the timing for sampling <0.063 mm diameter particles at 20 cm depth in the sediment cylinder at a liquid temperature of 20 °C is (in metric system):

$$t(d_p,z) = 0.2 / ((9.81 * (2650 - 1000) / (18 * 10^{-3})) * 0.000063^2) = 56 \text{ seconds} \quad \text{(A4)}$$

This corresponds to Sampling 1 as described in section 1.3 (see also current sampling times and depths in Annex 4b).

(b) Former sampling times and depths for pipette method

Former sampling times and depths for the pipette method are as described in Ljung (1987) and also relate to tables in Gee and Or (2002) (current sampling times/depths are given in brackets for comparison):

Sampling 1 (<0.063 mm): Formerly 30 s at 10 cm depth (currently 56 s at 20 cm).
Sampling 2 (<0.020 mm): Formerly 4 min 48 s at 10 cm (currently 4 min 38 s at 10 cm).
Sampling 3 (<0.006 mm): Formerly 53 min 20 s at 10 cm (currently 51 min 29 s at 10 cm).
Sampling 4 (<0.002 mm): Formerly 6 h at 7.5 cm (currently 5 h 48 min at 7.5 cm).

8.5 Annex 5 (see section 1.7): Using the program to calculate particle size distribution and loss on ignition for fine earth particles (d_p <2 mm)

The raw data (mass of different aliquots taken with the pipette and in the wet- and dry-sievings, and the loss on ignition as described in section 1.2-1.6), are entered into the program used for estimating particle size distribution.

Before introducing the measured data, some inputs must be made. First, in column A in the Excel file (see screen dump in Figure A7), numbers from 1 to X are entered for the batch of samples analysed. The designations of the samples are entered in columns B and C (both columns have the same information). In subsequent columns, the mass data from the crucibles with different fraction classes are entered (from left to right): the mass of the aliquots, starting with the results from Sampling 4 (column D), then Sampling 3 (E), Sampling 2 (F) and Sampling 1 (G), then the mass of the sand fractions from the wet and dry-sieving (columns H, I, J). Results from the mass of soil for loss on ignition estimations after drying to 105 ^{o}C and 550 ^{o}C are given in column K and L, respectively, and those from the dry-sieving of d_p >2 mm fractions in columns M, N and O.

A correction is needed due to the volume of liquid that the pipette can hold (columns D-G). The reason is that the calculation program is calibrated for a pipette with 10 mL volume, whereas the pipette used has a slightly smaller volume. Thus, when the pipette is replaced with a new one, this calibration factor has to be checked/modified again. The values for the aliquots are corrected by multiplying by a factor of 1.0298866 (see new values in yellow in the lower table in Figure A7, the rest are the same as in the upper table). The values in the lower table in Figure A7 are read by the program and used for the calculations. A correction factor for the mass of the dispersant agent (10.20 mg) is given as input to the program (as shown in the screen dump in Figure A15) to be deducted from the mass of each of the four pipette aliquot samples. The net mass after this is multiplied by 100 in order to account for all solution in the large cylinders. For an example of results, see Table 3 in section 1.7.

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P
1				g				g			glödförlust - tara		Sållning (ej tvättat)			
2	prov	Provbeteckning	Nivå								invägt	utvägt	<2	2 - 20	>20	
3	nr			d<0,002	d<0,006	d<0,02	d<0,06	0,06-0,2	0,2-0,6	0,2-2,0	g	g	g	g	g	
4	1	Soil A		0.0977	0.125	0.1497	0.1683	0.68	1.23	1.81	10.03	9.28	1097.73	3.7	0	
5	2	Soil B		0.1116	0.139	0.1631	0.1832	0.51	0.96	1.42	10.77	10.17	1260.09	4.5	0	
6	3	Soil C		0.0578	0.0714	0.0927	0.1167	4.09	2.77	4.08	10.39	9.98	1936.2	86.34	0	
7	4	Soil D		0.0649	0.0792	0.0998	0.1239	3.85	2.62	3.68	11.3	10.89	1730.16	65.42	31.26	
8	5	Soil E		0.0442	0.0553	0.0736	0.1	4.79	3.45	4.73	10.77	10.38	1381.85	102.52	16.2	
9	6	Soil F		0.0448	0.0568	0.0756	0.1031	4.73	3.38	4.72	10.67	10.31	1244.29	106.49	48.26	
10	7	Soil G		0.0723	0.0891	0.1092	0.1341	3.58	2.09	3.09	10.21	9.9	1210.71	35.99	21.99	
11																
12																
13																
14																
15	1.0298866															
16				g				g			glödförlust - tara		Sållning (ej tvättat)			
17	prov	Provbeteckning	Nivå								invägt	utvägt	<2	2 - 20	>20	
18	nr			d<0,002	d<0,006	d<0,02	d<0,06	0,06-0,2	0,2-0,6	0,2-2,0	g	g	g	g	g	
19	1	Soil A	Soil A	0.10062	0.128736	0.154174	0.17333	0.68	1.23	1.81	10.03	9.28	1098	4	0	
20	2	Soil B	Soil B	0.114935	0.143154	0.167975	0.188675	0.51	0.96	1.42	10.77	10.17	1260	5	0	
21	3	Soil C	Soil C	0.059527	0.073534	0.09547	0.120188	4.09	2.77	4.08	10.39	9.98	1936	86	0	
22	4	Soil D	Soil D	0.06684	0.081567	0.102783	0.127603	3.85	2.62	3.68	11.3	10.89	1730	65	31	
23	5	Soil E	Soil E	0.045521	0.056953	0.0758	0.102989	4.79	3.45	4.73	10.77	10.38	1382	103	16	
24	6	Soil F	Soil F	0.046139	0.058498	0.077859	0.106181	4.73	3.38	4.72	10.67	10.31	1244	106	48	
25	7	Soil G	Soil G	0.074461	0.091763	0.112464	0.138108	3.58	2.09	3.09	10.21	9.9	1211	36	22	
26																

Figure A7. Screen dump of an Excel file with example of data prepared for the program.

The screen dumps in Figures A8-A18 illustrate the procedures performed in the program. To open the program, choose “Läs in mekanalysdata från Excel och spara till disk” in the pop-up in Figure A8 (initial menu):

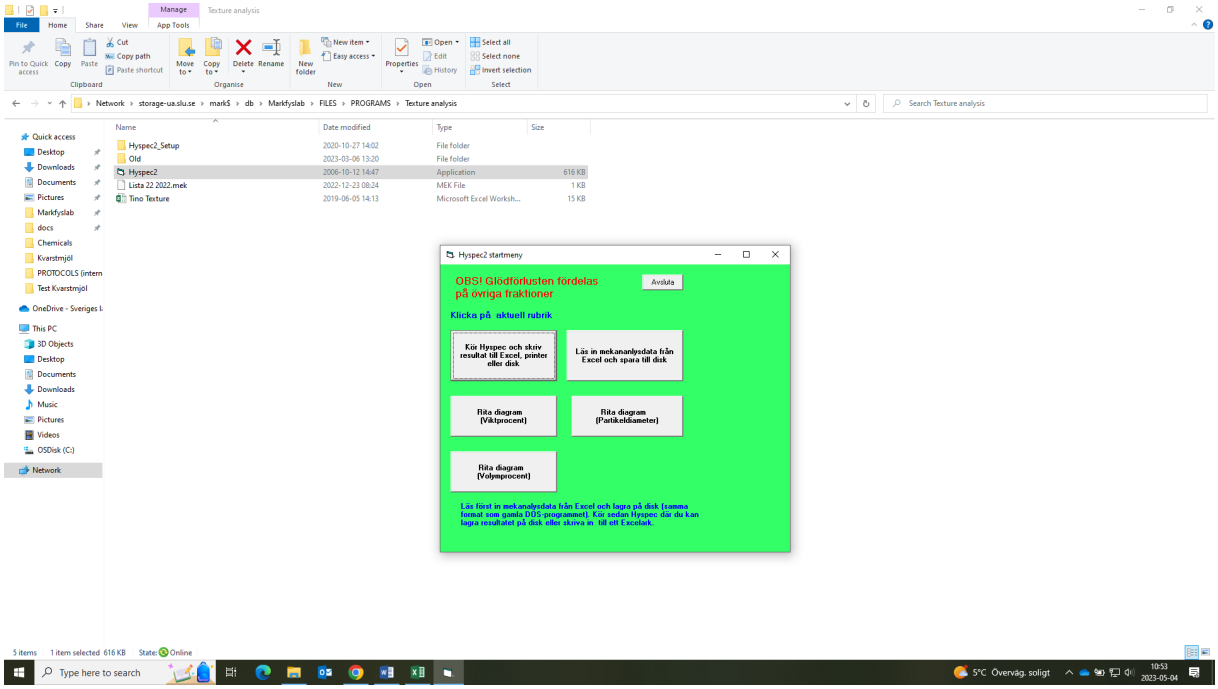


Figure A8. Screen dump of the opening page of the program (initial menu).

In the pop-up in Figure A9, tell the program where the data are in the Excel file: number of the first row “Indata startar på rad”, the last row “och slutar på rad”, and the name of the sheet “Namn på Excelarket”. Choose “0.1130” in the red box and click “Läs in från Excel” to continue:

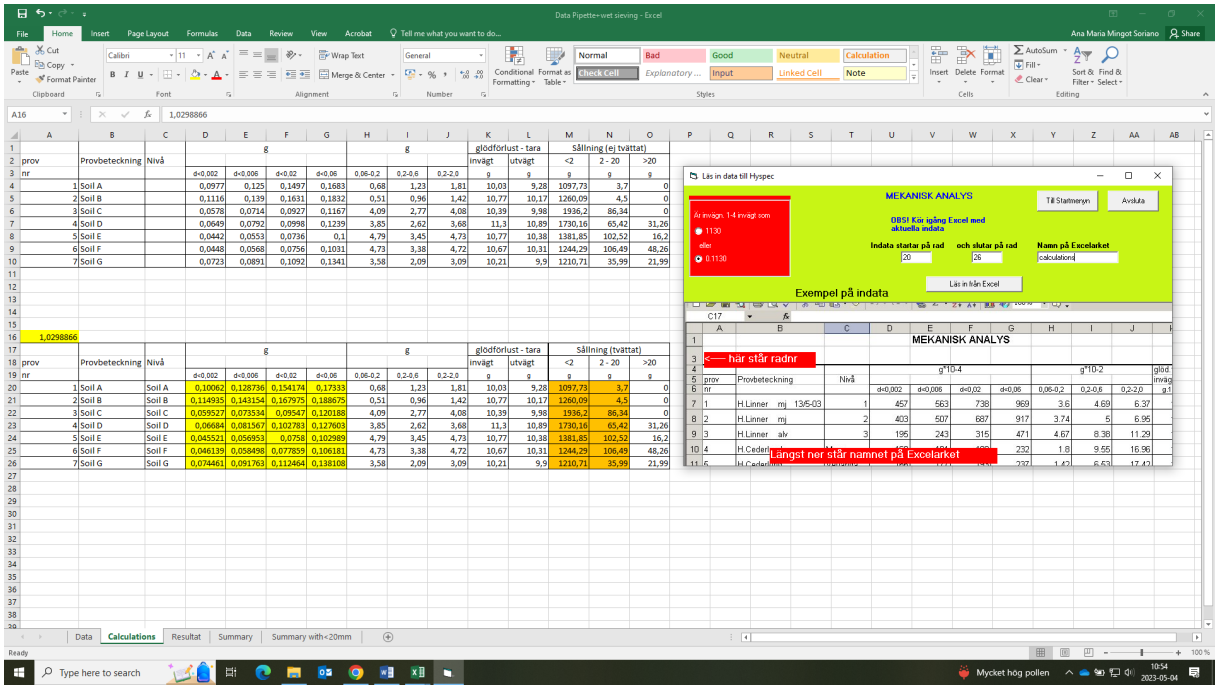


Figure A9. Screen dump of detecting an Excel file with examples of data entered.

Confirm that the data in the pop-up in Figure A10 are correct and click “Lagra data”:

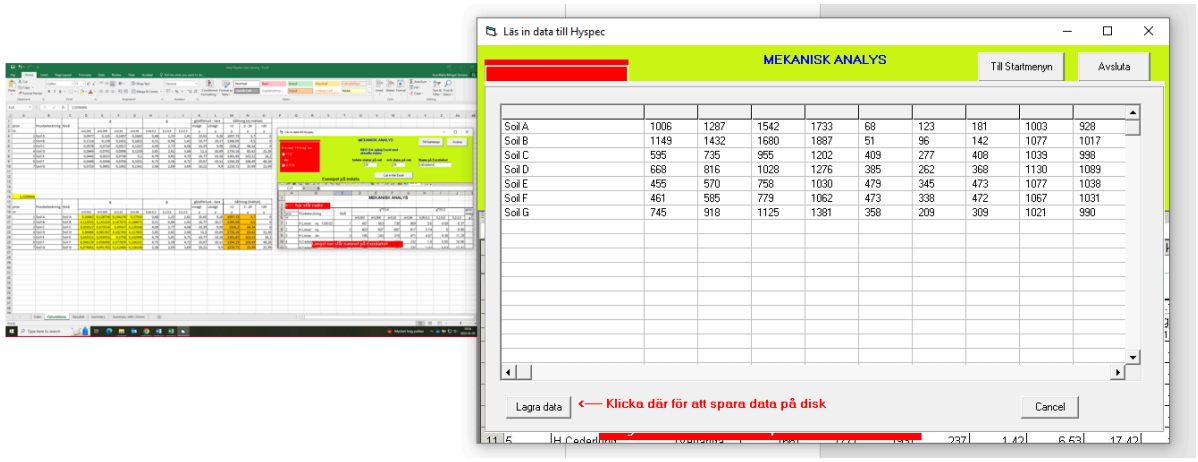


Figure A10. Screen dump of recalculated mass fraction values from an Excel file with examples of data entered.

In the pop-up in Figure A11, tell the program where the data come from, when the new file (for calculations) is created, and the name of the new file.

Note: The program has 20 grams of sample by default (at “Vikt (g)”). If all the samples have another mass (e.g. 30 grams, because they contain very much sand), it is possible to change the default value at this step.

After a message saying “Lagrat data på disk”, click “OK” and continue:

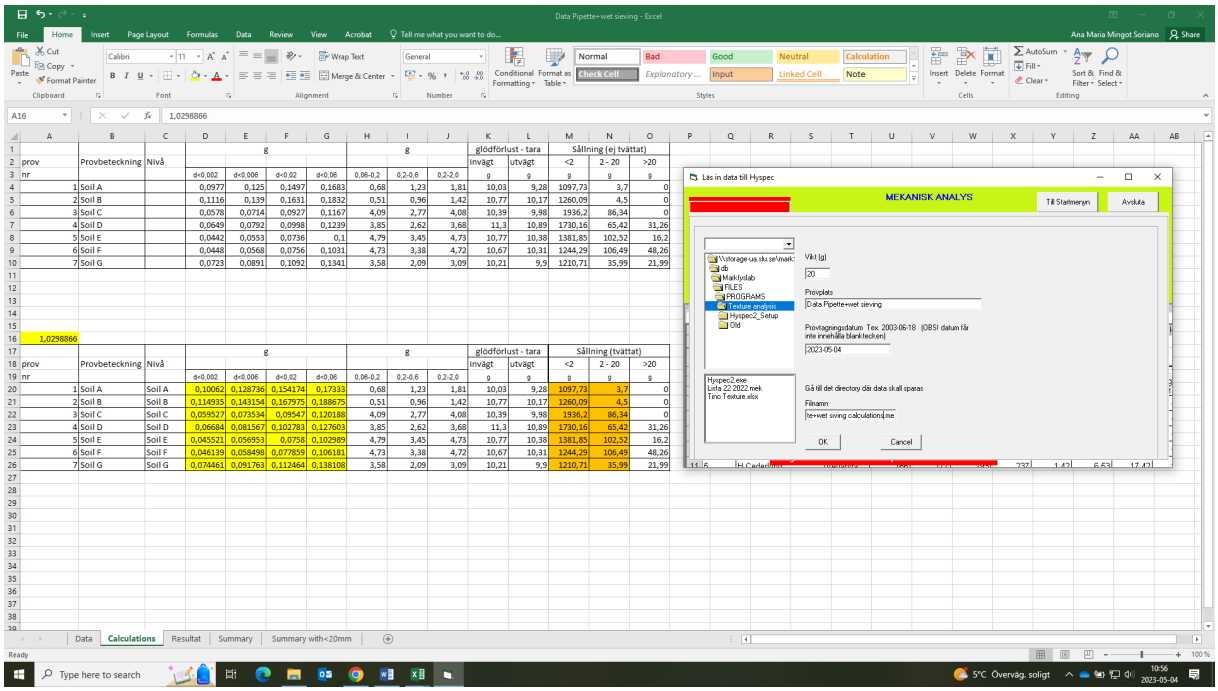


Figure A11. Screen dump of pop-up with entry of further specifications (sample mass, and in- and output file information).

In the pop-up in Figure A12, click “Till Startmenyn” to go back to the initial menu:

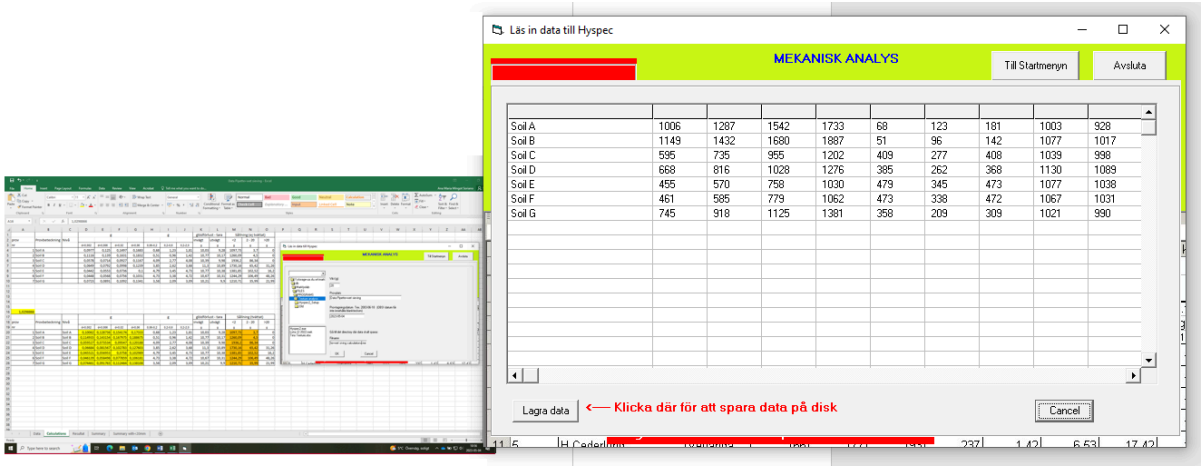


Figure A12. Screen dump on returning to the initial menu.

In the pop-up in Figure A13, choose “Kör Hyspec och skriv resultat till Excel, printer eller disk”:

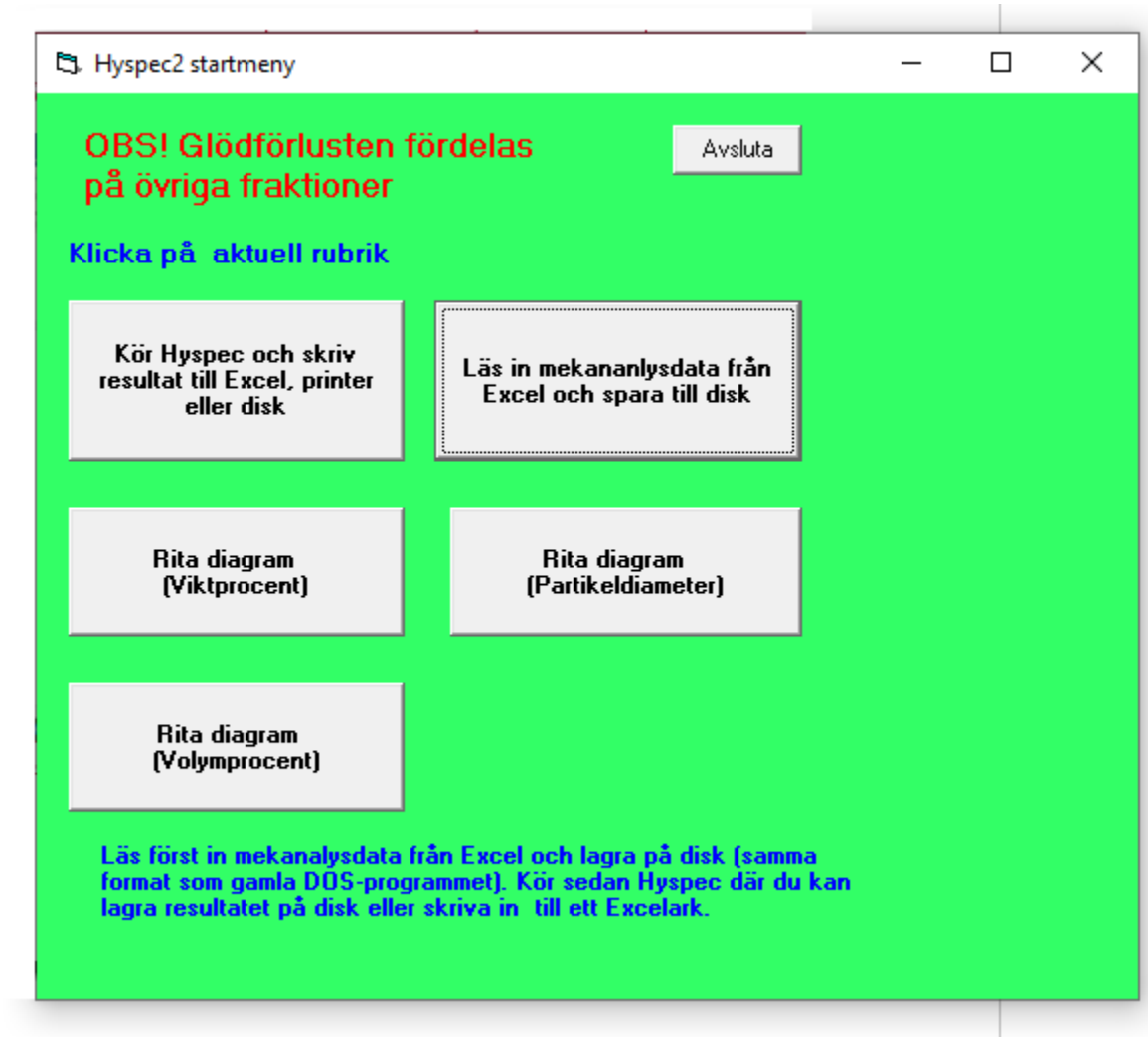


Figure A13. Screen dump of the Hyspec initial menu.

In the pop-up in Figure A14, choose the created new file (for calculations) in the list of files to the left and click on “Läs in resultat till Excel”:

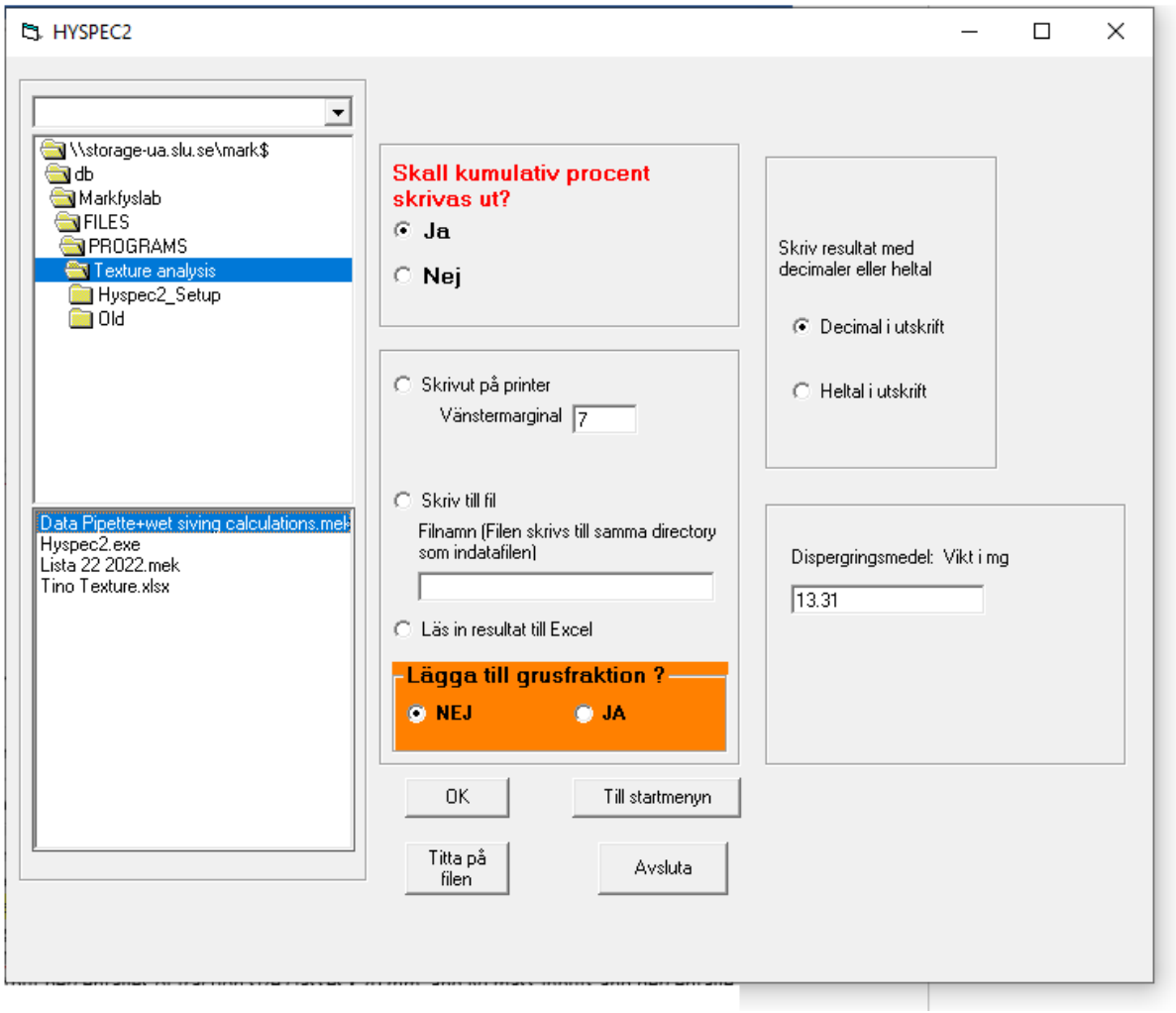


Figure A14. Screen dump of Hyspec data entry information (1).

In the pop-up in Figure A15, fill in the information regarding where to write the results (row number of the first row of fine earth fraction d_p <2 mm "Ange radnr där resultat skall börja skrivas" and, if desired, the gravel fraction "Om grusfraktion skall skrivas ut", and the name of the sheet "Excelarkets namn") and the dispersant agent mass "Dispergeringsmedel" (10.20 mg for the new dispersant). Click "OK":

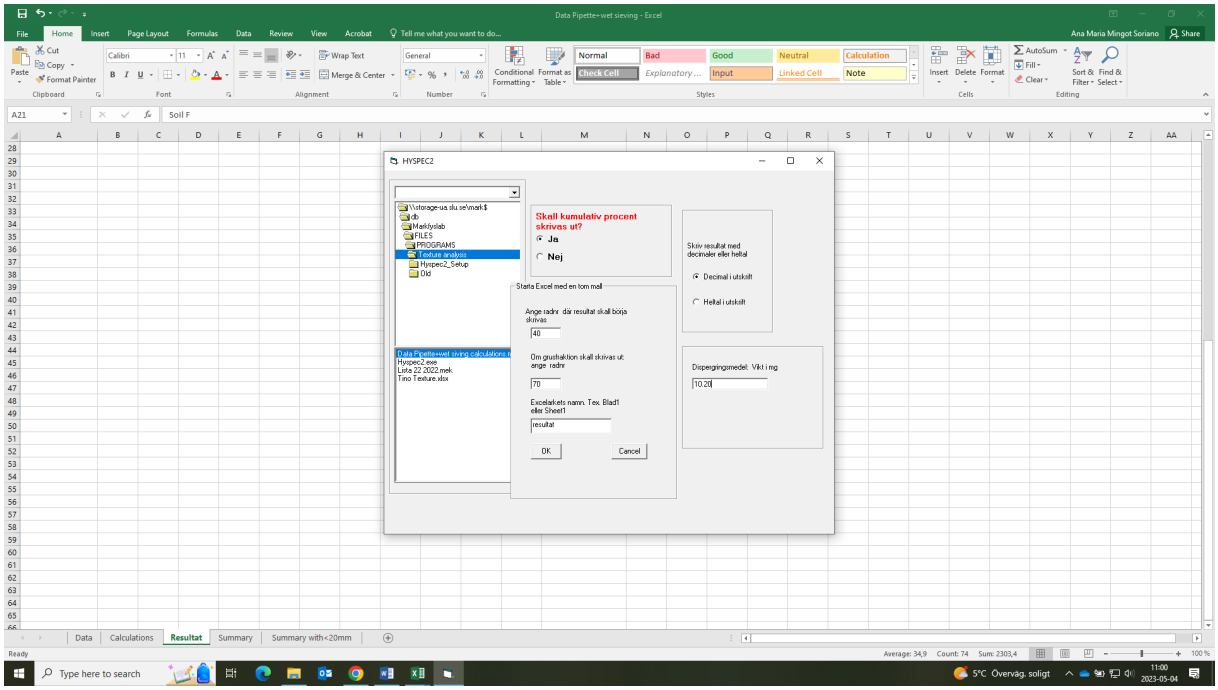


Figure A15. Screen dump of Hyspec data entry information (2).

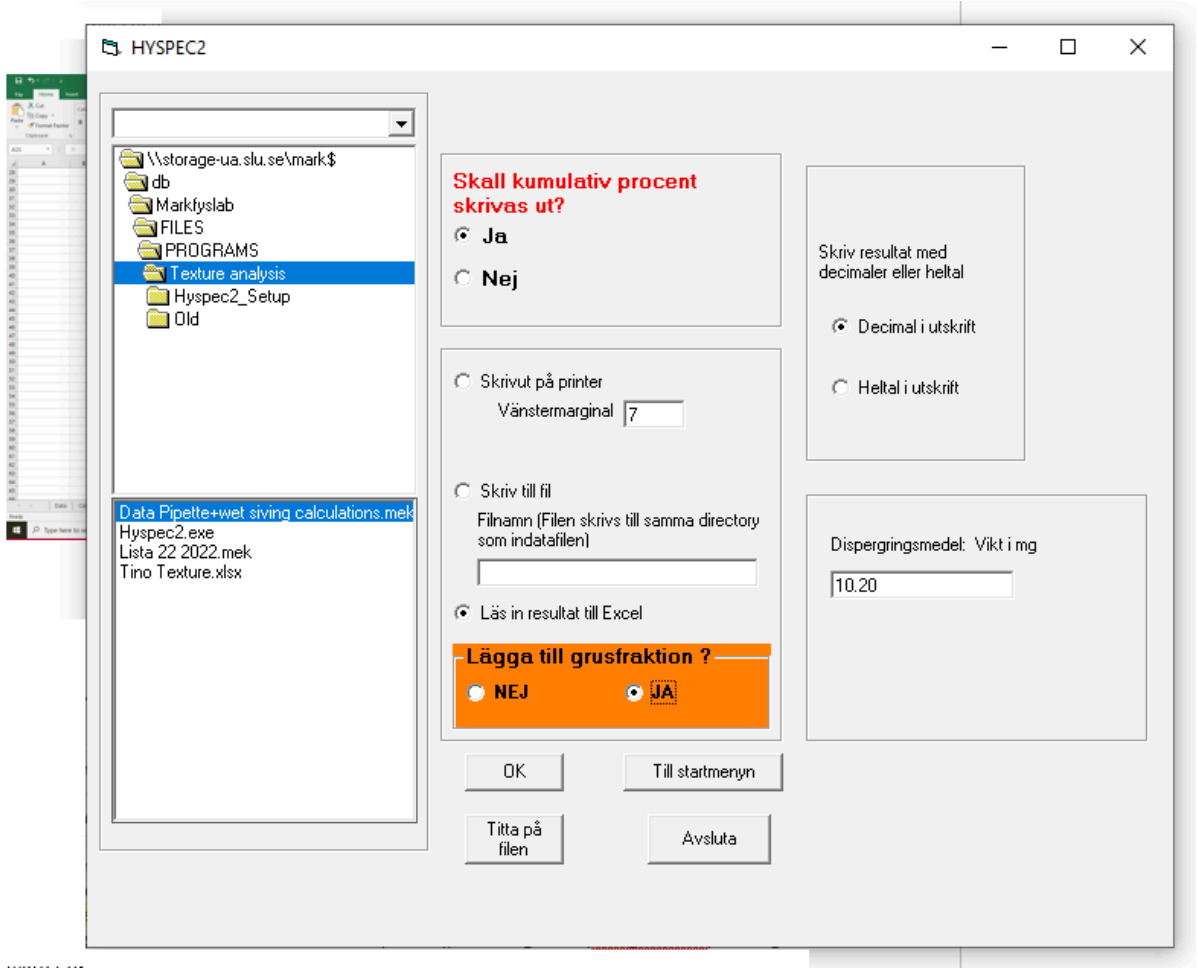


Figure A16. Screen dump of Hyspec data entry information (3).

If there are particles with d_p 2-20 mm (see also Annex 6), click “JA” on the orange area and then “OK” in the pop-up in Figure A16. If not, click “NEJ” and “OK”.

In the pop-up in Figure A17 (in cases when there are particles with d_p 2-20 mm as defined with “JA” on the orange area in Figure A16), write the mass in grams (no decimals) of the fractions d_p <2 mm and 2-20 mm, the latter in the >2 mm column (*Note: The analysis of any d_p >20 mm fraction, calculated separately in Excel, is described in Annex 6*). Click “OK” and close the program:

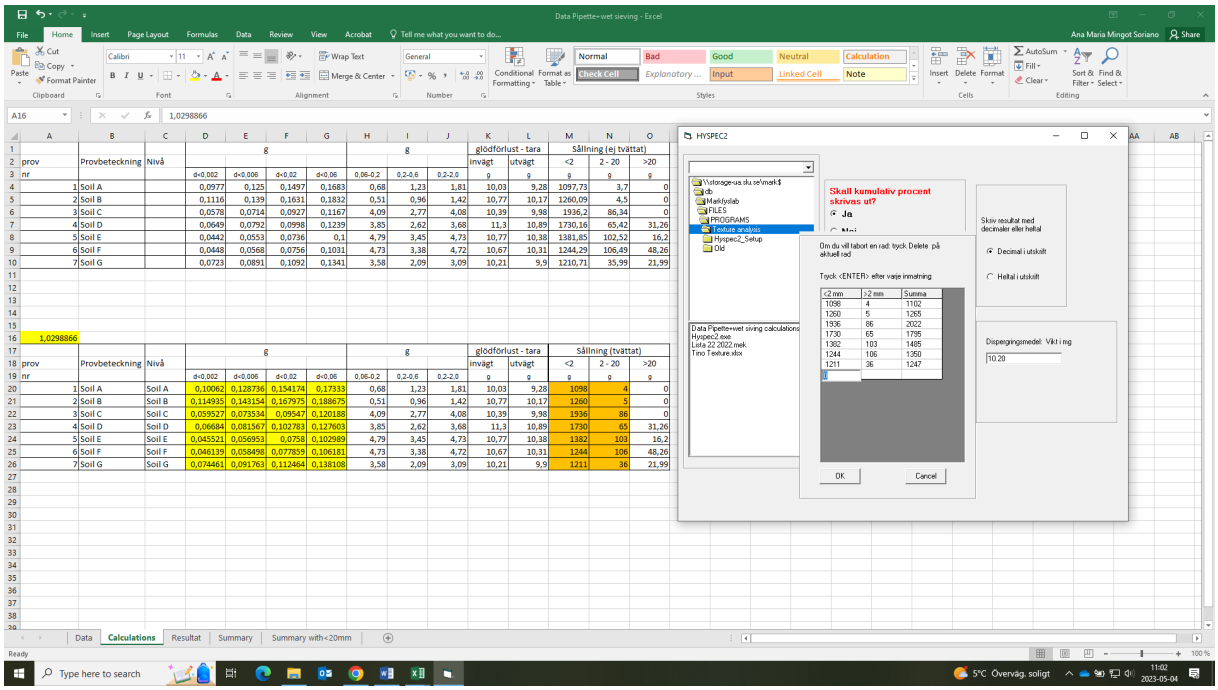
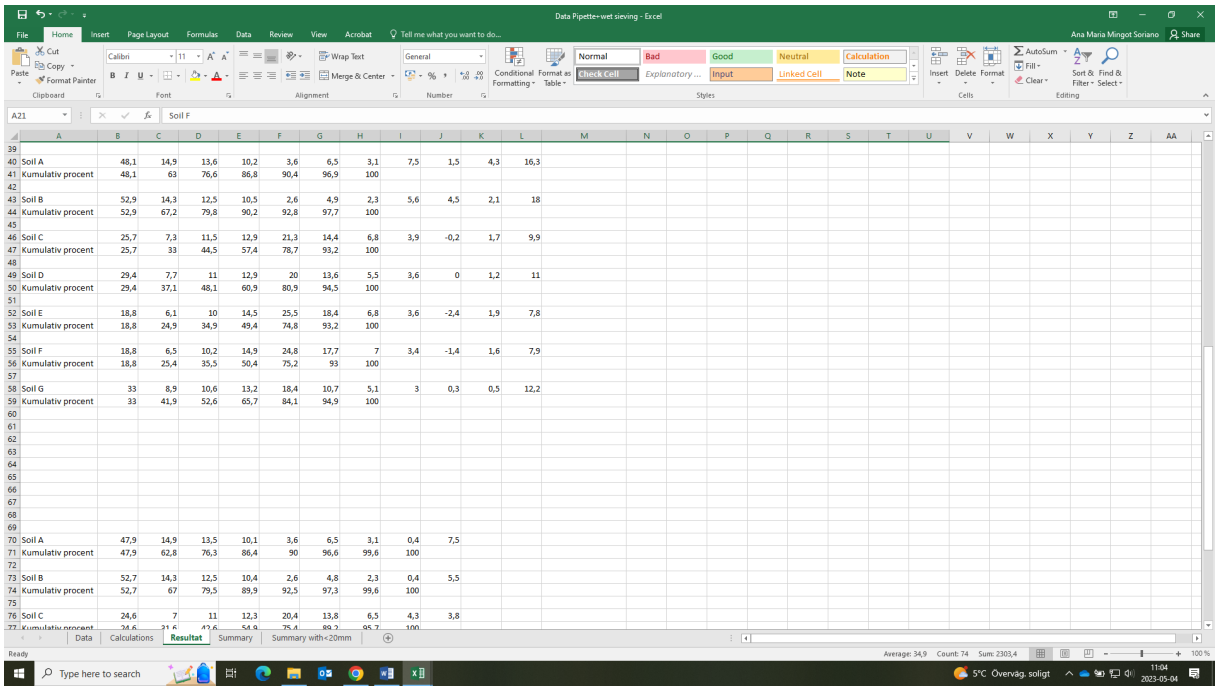


Figure A17. Screen dump of Hyspec data entry information (4).

The results will appear where the program was told to write them as shown in Figure A18 (see also Table 3 in section 1.7 for the d_p <2 mm fractions and in Annex 6 for the d_p >2 mm fractions):



40	Soil A	48,1	14,9	13,6	10,2	3,6	6,5	3,1	7,5	1,5	4,3	16,3
41	Kumulativ procent	48,1	63	76,6	86,8	90,4	96,9	100				
43	Soil B	52,9	14,3	12,5	10,5	2,6	4,9	2,3	5,6	4,5	2,1	18
44	Kumulativ procent	52,9	67,2	79,8	90,2	92,8	97,7	100				
46	Soil C	25,7	7,3	11,5	12,9	21,3	14,4	6,8	3,9	-0,2	1,7	9,9
47	Kumulativ procent	25,7	33	44,5	57,4	78,7	93,2	100				
49	Soil D	29,4	7,7	11	12,9	20	13,6	5,5	3,6	0	1,2	11
50	Kumulativ procent	29,4	37,1	48,1	60,9	80,9	94,5	100				
52	Soil E	18,8	6,1	10	14,5	25,5	18,4	6,8	3,6	-2,4	1,9	7,8
53	Kumulativ procent	18,8	24,9	34,9	49,4	74,8	93,2	100				
55	Soil F	18,8	6,5	10,2	14,9	24,8	17,7	7	3,4	-1,4	1,6	7,9
56	Kumulativ procent	18,8	25,4	35,5	50,4	75,2	93	100				
58	Soil G	33	8,9	10,6	13,2	18,4	10,7	5,1	3	0,3	0,5	12,2
59	Kumulativ procent	33	41,9	52,6	65,7	84,1	94,9	100				
70	Soil A	47,9	14,9	13,5	10,1	3,6	6,5	3,1	0,4	7,5		
71	Kumulativ procent	47,9	62,8	76,3	86,4	90	96,6	99,6	100			
73	Soil B	52,7	14,3	12,5	10,4	2,6	4,8	2,3	0,4	5,5		
74	Kumulativ procent	52,7	67	79,5	89,9	92,5	97,3	99,6	100			
76	Soil C	24,6	7	11	12,3	20,4	13,8	6,5	4,3	3,8		

Figure A18. Screen dump of the Hyspec results page.

8.6 Annex 6 (see section 1.7): Procedure for particles with d_p >2 mm in soil sample

When there are particles with d_p >2 mm, the proportions of fine earth (d_p <2 mm), gravel (d_p 2-20 mm) and cobbles (d_p >20 mm) may be determined (see Figure A19). Thus, the program recalculates the proportion for each fraction class taking into account also the particles with d_p 2-20 mm. For this, the mass of fine earth (d_p <2 mm) and the mass of gravel (d_p 2-20 mm) must be entered manually into the program (see Figures A16 and A17). The proportion of cobbles (d_p >20 mm) is calculated separately in Excel and thereafter added to the results. The tables in Figure A19 illustrate two samples with: (i) mass inputs for air-dried water content (*vattenhalt*), loss on ignition (*glödförlust*) and the particle size fraction classes from dry-sieving (*sållning*) d_p <2, 2-20 and >20 mm (from Table 2 in section 1.7, soils F and G); (ii) output percentages of fine earth fraction size classes (d_p <2 mm), loss on ignition (*glödgn.förlust*) and organic matter (*mullhalt*) (the latter estimated from loss on ignition) (from Table 3 in section 1.7, soils F and G); (iii) output percentages of fraction size classes d_p <20 mm; and (iv) mass inputs and percentage outputs for dry-sieving fraction size classes d_p <2, 2-20 and >20 mm.

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
1															
2	**MEKANISK ANALYS (glödgningsförlust)**														
3			glödförlust				glödförlust - tara			Vattenhalt	Glödförlust	Sållning (ej tvättat)			
4	prov	Provbetec	tara dege	före tork	105°	550°	före tork	105°	550°			<2	2 - 20	>20	
5	nr		g	g	g	g	g	g	g	%	%	g	g	g	
6	6	Soil F	38.44	49.27	49.11	48.75	10.83	10.67	10.31	1.5	3.4	1244.29	106.49	48.26	
7	7	Soil G	36.95	47.41	47.16	46.85	10.46	10.21	9.9	2.4	3.0	1210.71	35.99	21.99	
8															
9	**Mekanisk analys (fraktionerna angivna i %)**														
10															
11	Finjord <2mm	Ler	Fin mjäla	Grov-mjäla	Fin-mo	Grov-mo	Mellan-sand	Grov-sand	Glödgn.	Fel%	Mull-halt				
12			0,002-	0,006-	0,02-	0,06-	0,2-	0,6-	förlust						
13		d<0,002	0,006	0,02	0,06	0,2	0,6	2	%						
14	Soil F	18.8	6.5	10.2	14.9	24.8	17.7	7	3.4	-1.4	1.6				
15	Kumulativ procent	18.8	25.4	35.5	50.4	75.2	93	100							
16															
17	Soil G	33	8.9	10.6	13.2	18.4	10.7	5.1	3	0.3	0.5				
18	Kumulativ procent	33	41.9	52.6	65.7	84.1	94.9	100							
19															
20															
21	Grusfraktion <20mm	Ler	Fin-mjäla	Grov-mjäla	Finmo	Grovmo	Mellan-sand	Grov-sand	Grus						
22			0,002-	0,006-	0,02-	0,06-	0,2-	0,6-	2,0-						
23		d<0,002	0,006	0,02	0,06	0,2	0,6	2	20						
24	Soil F	17.4	6	9.4	13.7	22.9	16.3	6.5	7.9						
25	Kumulativ procent	17.4	23.4	32.7	46.4	69.3	85.7	92.1	100						
26															
27	Soil G	32.1	8.6	10.3	12.8	17.9	10.4	5	2.9						
28	Kumulativ procent	32.1	40.7	51.1	63.8	81.7	92.1	97.1	100						
29															
30															
31	Sållning	<2mm	2-20mm	>20mm	summa	<2mm	2-20mm	>20mm							
32		g	g	g	g	%	%	%							
33	Soil F	1244.29	106.49	48.26	1399.04	88.9	7.6	3.4							
34	Soil G	1210.71	35.99	21.99	1268.69	95.4	2.8	1.7							
35															

Figure A19. Recalculation for particles >2 mm.

8.7 Annex 7 (see section 4): Special sandboxes

At the Soil Physics Laboratory there are two special sandboxes (Figure A20). These sandboxes were constructed at the former Soil Science Department, because soil water retention properties were generally determined simultaneously at more suction steps than today. The conventional sandboxes were used for higher suctions (up to 1 m) (see section 4.2) whereas these special sandboxes were used only for low suctions (i.e. near zero to 7.5 cm).

Figure A20. Special sandboxes.

These special boxes contain a porous plate with pore size equal to sand, instead of a sand bed and cloth. Otherwise, the samples are prepared in the same way: soil cylinder with a yellow lid on top (to prevent evaporation from the surface), filter paper and cloth on the bottom, fastened with a rubber band (see section 4.1).

Maintenance:
When these special sandboxes are not in use, they must be kept closed and dry (no water inside). Once measurements are finished, the box must be cleaned. Water is removed and the porous plate is brushed without soap, only by rinsing. No products to remove algae or fungi have been needed so far.

Before experimental run:
Before using the special sandbox, it needs to be moistened using boiled tap water that has cooled slightly (but is still warm). If the porous plate is initially completely dry, it may take one day to get it wet again, but the main problem may be air bubbles in the pipes. Water is added to the box and allowed to drain for two days, to ensure that the system (porous plate and pipes) is moist.

Experimental run:
Before placing the samples in the moistened special sandbox, water is added and a listening check is made to ensure that there are no more bubbles coming from the porous plate.
Then the samples are placed on the porous plate, the box is closed with the main lid, a bucket is placed to collect the water and the valve is opened to drain. The subsequent procedure is the same as for the conventional sandboxes described in section 4.2.

8.8 Annex 8 (see section 5.3): (a) Theory: calculation of saturated hydraulic conductivity and (b) Parameter settings

(a) Theory: calculation of saturated hydraulic conductivity from water flow volume and time, hydraulic head difference and sample area and height (Darcy´s law)

Applied to water flow through a cylindrical soil sample in a permeameter, Darcy´s law, adapted from Darcy (1856), describes how the volume of the measured flow (V, mL or cm^3) by time unit (t, hour) is related to the saturated hydraulic conductivity (K_s, cm $hour^{-1}$), the area of the sample (A, cm^2) and the hydraulic gradient (cm cm^{-1}, i.e. dimensionless), the latter being the total hydraulic head (water pressure head plus gravitational head) difference (dH, cm) acting on the sample divided by the height of the sample (dx, cm), so that:

$$V / t = -K_s * A * (dH/dx) \quad \text{(A5)}$$

The equivalent expression for calculation of the saturated hydraulic conductivity K_s is:

$$-K_s = (V / t) / (A * (dH/dx)) = (V * dx) / (t * A * dH) \quad \text{(A6)}$$

A correction factor ($Tkorr$) to adjust for water viscosity variations (caused by water temperature variations) is inserted in the equation so that:

$$-K_s = (V * Tkorr * dx) / (t * A * dH) \quad \text{(A7)}$$

(b) Parameter settings related to the permeameter measurements/calculations of saturated hydraulic conductivity in section 5.3

In the equation (A7) used to estimate the saturated hydraulic conductivity K_s, i.e. $-K_s = (V * Tkorr * dx) / (t * A * dH)$, the following specifications and relations to the measurement set-up described in section 5.3 are used:

- V is the actual measured volume retained in the graduated cylinder below the soil sample (see Figure 47) during a certain time t.
- $Tkorr = 1.697801 - (0.04481396 * Temp) + (0.0005085822 * Temp^2)$, where $Temp$= temperature in gutter water during experimental run in ^oC.
- dx is the height of the cylinder core soil sample, which in our laboratory is generally 5 or 10 cm.
- t is the time needed to gather the measured volume V retained in the graduated cylinder below the soil sample.
- A is the surface area of the cylinder core soil sample, which is calculated by the general formula for circle area: our cylinders have inner diameter of 7.2 cm, and thus $A = \pi * (7.2 / 2)^2$.

- *dH* is equal to the difference between the level of the incoming water, as defined by the steady water level controlled by the water intake overflow in the water gutters, and the level of the outflowing water through the hole on the overflow container surrounding the soil cylinder sample (see Figures 40-41 in section 5.3).

Figure A21 shows a screen dump of an example of Excel file used for calculations (the symbol K in the screen dump is identical to $-K_s$ in the equation A7 above):

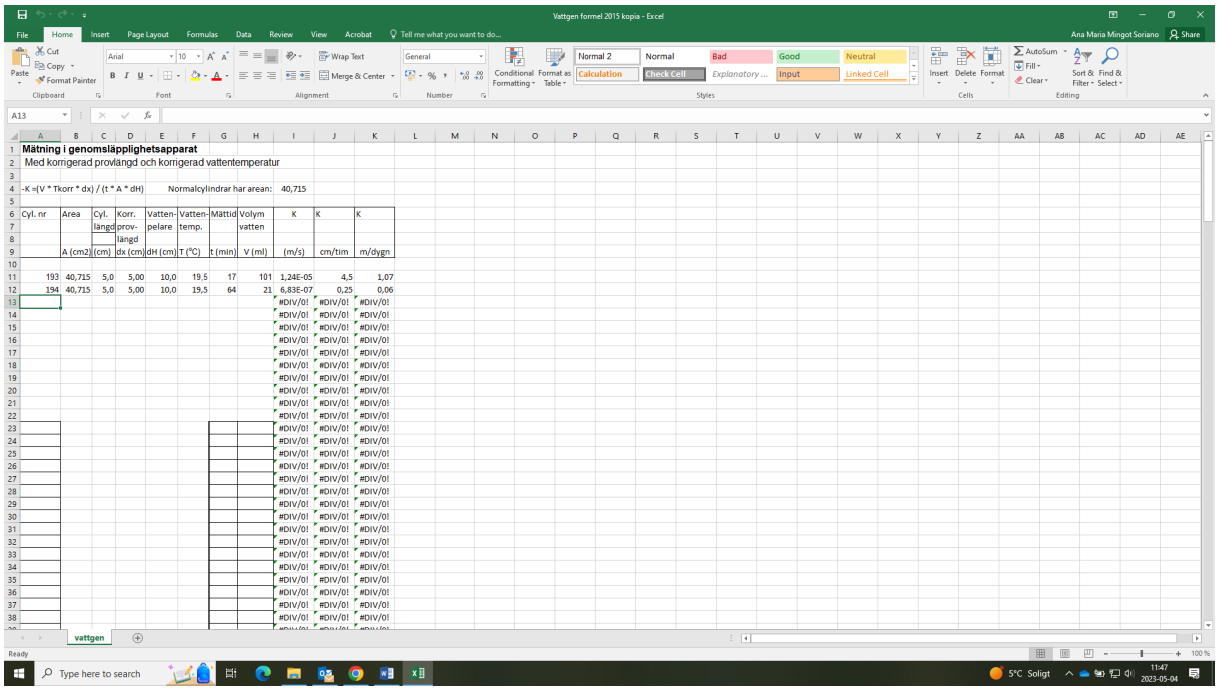


Figure A21. Screen dump of an Excel file used for calculation of saturated hydraulic conductivity.

9. Glossary: Vocabulary English - Swedish

Actual field soil water content	- Aktuell fältvattenhalt
Dry bulk density	- Torr skrymdensitet (tidigare torr volym-vikt)
Gravimetric soil water content	- Gravimetrisk vattenhalt (vattenhalt i viktsprocent)
Loss on ignition	- Glödförlust
Organic matter content	- Mullhalt
Particle density	- Kompaktdensitet (tidigare specifik vikt)
Particle size distribution (clay, fine silt, medium silt, coarse silt, fine sand, medium sand, coarse sand, gravel, cobble)	- Kornstorleksfördelning (ler, finmjäla, grovmjäla, finmo, grovmo, mellansand, grovsand, grus, sten)
Porosity	- Porositet
Saturated hydraulic conductivity	- Mättad hydraulisk konduktivitet (vattengenomsläpplighet)
Shrinkage	- Krympning
Volumetric soil water content	- Vattenhalt i volymsprocent
Water retention properties	- Vattenhållande förmåga
Wilting point	- Fysikalisk vissningsgräns