# Laboratory and Field Exercises Merging Measurement and Modelling of Soil Physics

# **Transport Processes**

2023

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# 1. Site descriptions

investigated sites									
	Clay	Silt	Sand	Organic carbon	Soil type	Bulk density			
	%	%	%	%	(USDA)	g/cm <sup>3</sup>			
Jynde- vad	4.7	4.4	90.8	3.0	Sand	1.44			
Estrup	13.0	28.5	58.6	2.2	Sandy loam	1.38			





# Jyndevad

The sampling site located in southern Denmark covers a cultivated area of about 2.39 ha (184 m x 135 m) and exhibits a slope of 0–1%. In this particular area, there are abundant Quaternary deposits of late glacial freshwater sands. The soil at the site contains of coarse-grained sand and has a natural organic matter gradient with organic matter content increasing from north to south. The three soil profiles at the site have been classified as Typipodsols. equivalent to a Humic Psammentic Dystrudept (Lindhardt et al., 2001; Masis-Melendez et al., 2014).

# Estrup

The sampling site located in Estrup in central Denmark covers a cultivated area of about 1.26 ha (110 m x 130 m). The site has a complex geological structure comprising a clay till core with deposits of different age and composition such as post-glacial peat, meltwater sand as well as clay and gyttja. The soil profiles developed in the clay till are classified as Pseudogleytypilessive (Lindhardt et al., 2001).

## References

- Karup, D., Moldrup, P., Paradelo, M., Katuwal, S., Norgaard, T., Greve, M. H., and de Jonge, L. W. (2016). Water and solute transport in agricultural soils predicted by volumetric clay and silt contents. *Journal of Contaminant Hydrology* **192**, 194-202.
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- Masis-Melendez, F., Deepagoda, T. K. K. C., de Jonge, L. W., Tuller, M., and Moldrup, P. (2014). Gas diffusion-derived tortuosity governs saturated hydraulic conductivity in sandy soils. *Journal of Hydrology* **512**, 388-396.

# 2. Soil water retention curve

## **Background:**

### Soil-water retention

The relationship between the soil moisture content and soil-water potential is called the soil-water characteristic curve or soil-water retention curve. It specifies how much water the soil retains at a given soil-water potential. This relationship is unique for each soil and must be measured experimentally. Together with the soil hydraulic conductivity, the soil-water retention curve is used extensively in modelling water flow through the soil.

What is the soil-water retention curve?

In saturated soil in equilibrium with free water, the actual pressure is atmospheric, and the hydrostatic pressure and suction are zero. At this pressure, the soil-water content is approximately equal to the soil porosity and the soil is called saturated (water content at saturation:  $\theta_s$ ). Suppose a slight suction, i.e. a water pressure slightly below atmospheric pressure, is applied to a saturated soil. In that case, no water outflow occurs until the suction is increased above a specific value. At this particular value, the largest soil pores begin to empty; the critical level of suction is called the air entry value (of suction),  $-\psi_e$ . Its value is generally around  $-10 \text{ cm H}_2O$  for many soils. It is often relatively small in coarse-textured and well-aggregated soils. However, in such soils, the pores are often nearly uniform in size, and these soils often exhibit air-entry phenomena more distinctly defined than for fine-textured soils. As more energy is applied and the suction is further increased, more water drains out of the soil, and more relatively large pores, which cannot retain water against the suction applied, will empty.

The soil water retention curve is measured in the laboratory using various set-ups (sandboxes, pressure plates and potentiometer), each applicable at different ranges of negative pressure heads. Sandboxes, vacuum chambers and pressure plates are used on the wet end of the soil-water retention curve (-10 to  $-15,000 \text{ cm H}_2\text{O}$ ), whereas the potentiometer measures the dry part of the soil-water retention curve.

### Procedure

### Wet part of the soil water retention curve

Before the course, all the samples were water-saturated over three days by slowly raising the water level to the rim of the ring. Afterwards, to the samples were saturated to  $-100 \text{ cm H}_2\text{O}$ .

- 1. Weigh out the samples.
- 2. Create a water film on the sand by opening the valve on the water supply container.
- 3. Put the samples in the sandbox and establish capillary contact between the sand surface and soil sample by carefully pressing the rings onto the wet surface.
- 4. Saturate the samples slowly by raising the water level to the rim of the ring in sandboxes, and keep it saturated for three days (duration is dependent on soil type and organic matter contents).
- 5. Drain the samples to a matric potential of -100 cm water column, as shown below.



- 6. After a few days, the samples are equilibrated with the selected matric potential; take the samples out of the sandboxes and record the weight on the laboratory scale connected to the lab computer.
- After finishing the measurements on these soil samples, put them in the 105°C oven to get the soil's dry weight.

#### Dry part of soil water retention curve (WP4C Dewpoint Potentiometer)

### Introduction

WP4 is an instrument that measures the potential energy status of water per unit mass of water in the soil; specifically measures the sum of osmotic and matric potentials in a sample. The total water potential of a soil sample is the sum of four components potentials gravitational, matric, osmotic and pressure. WP4 works by measuring the vapour pressure of air (p) in the headspace of a chamber in equilibrium with the saturation vapour pressure ( $p_0$ ) from the liquid phase water of the soil sample. During the analysis, the soil sample is placed into a cup, which is sealed against a sensor block. A dew point sensor measures the dew point temperature of the air, therefore, the vapour pressure of air is computed as the saturation vapour pressure at the dew point temperature. An infrared thermometer measures the temperature in the sample to calculate the saturation vapour pressure in equilibrium. The relationship between the sample's water potential ( $\Psi$ ) and the vapour pressure of the air is:

$$\Psi = \frac{RT}{M} \ln \frac{p}{p_0}$$

Where *p* is vapour pressure of the air and  $p_0$  is the saturation vapour pressure at sample temperature, R is the gas constant (8.31 J/mol K), T is the Kelvin temperature of the sample, and M is the molecular mass of water.

### Procedure

 Before the start of the measurement turn on the instrument at least 30 min before to provide a warmup period. The ON/OFF switch is located on the lower left corner of the WP4's back panel. The LCD panel will show all the readings in cero value.



2. Verify the calibration using using a 0.5 molal KCl solution in a sample cup; filled just at the half, place it in the WP4's drawer. Carefully slide the drawer to close it. Keep the drawer knob in OPEN/LOAD position and press the key bottom down on the right of the front panel to display the sample equilibration screen. Wait for the temperature to equilibrate at the sample temperature menu (Ts-Tb). Before the measurement, the temperature of the sample (Ts) must be lower than the sensor block (Tb) to avoid condensation of water.

Ts =	24.9
Ts - Tb =	-0.07

- 3. Once Ts-Tb is < 0, turn the drawer knob to the READ position, the instrument will beep once and the green light will flash once to indicate that the reading has started. Wait until the green LED flash is constant, the beep sounds again and the water potential is displayed on the screen. Repeat one more time, the second reading of the standard ± 0.1pF of the correct reading.</p>
- 4. Turn the sampler drawer knob containing the standard to the OPEN/LOAD position and pull carefully avoiding any spill inside the instrument. Place the soil into a sampler cup until half of the volume. Slide the drawer to close it and check the Ts-Tb. Once Ts-Tb < 0, turn the drawer knob to the READ position. Take two readings for each sample.</p>
- 5. The gravimetric water content is usefully related to the water potential. Therefore, for each sample, weigh an empty aluminium tray and place the soil sample, weigh again to get the weight of soil+water. Put the aluminium tray into the oven dryer at 105 °C for 24 hours and get the weight of dry soil.

The operating range of the WPC is from pF 3.5 to 6.5 or -0.3 to -300 MPa

### Reference

Operator's manual WP4 Dewpoint Potentiometer. 1998-2007. Version 5.Decagon Devices, Inc.

# 3. Gas diffusion

## Background

The air-filled pore space in soil contains multiple gases, including oxygen, which is essential for the respiration of plant roots and soil organisms. In addition, this air space also contains carbon dioxide as a product of the respiration of plant roots and soil organisms.

According to the kinetic theory of gases, gas molecules in soil are in continuous thermal motion. In soil, a concentration gradient causes a net movement of molecules from high concentration to low concentration, which causes the movement of gas by diffusion. Numerically this movement is explained by Fick's laws of diffusion. Soil-gas diffusion in natural, undisturbed soil at different water contents depends on soil texture and structure, pore size distribution, pore connectivity and tortuosity.

## Principles of the measurement

Gas diffusivity is measured by a non-steady-state method (Rolston and Moldrup, 2002).  $O_2$  is chosen as the diffusing gas. The diffusion of  $O_2$  through the soil sample is registered by measuring the  $O_2$  concentration through the chamber over time.

## Theory

The diffusivity through the soil sample is induced by a non-steady state diffusion gradient, which can be explained by Fick's second law:

$$\frac{dC}{dt} = \frac{D_p}{\varepsilon} \frac{d^2 C}{dx^2}$$
[1]

where  $\varepsilon$  is the air-filled pore volume,  $D_p$  is the diffusion coefficient, C is the gas concentration, t is time, and x is the length.

Fick's first law says that

$$\frac{dq}{dt} = -D_p A \frac{\Delta C_t}{h_s}$$
[2]

where q is the volume of gas, A is the area of the soil sample,  $\Delta C_t$  is the concentration gradient through the soil sample, and  $h_s$  is the height of the soil sample.

To calculate  $D_p$ , we can solve Eq. [1] analytically (Rolston and Moldrup, 2002), or use Eq. [2] following the simple method described below.

The volume of gas diffusing into the diffusion chamber can also be expressed as

$$\frac{dq}{dt} = \frac{d(\Delta C_t)}{dt} h_c A$$
[3]

where  $h_c$  is the height of the diffusion chamber

Combining Eq. [2] and [3] gives

$$D_p dt = -h_s h_c \frac{d(\Delta C_t)}{\Delta C_t}$$
[4]

Integration gives ( $\Delta C_t = \Delta C_0$  for t = 0,  $\Delta C_t = \Delta C_t$  for t = t)

$$\ln\left(\frac{\Delta C_t}{\Delta C_0}\right) = -\frac{D_p}{h_s h_c} t \quad \Leftrightarrow \quad \ln\left(\frac{\Delta C_t}{\Delta C_0}\right) = K t$$
<sup>[5]</sup>

which expresses a linear relationship between the logarithm of the concentration difference and time.

Therefore, according to Eq. [5],  $D_p$  can be calculated by

$$D_p = -h_s h_c K$$
 [6]

### Exercise

1. Open the diffusion chambers and remove the blind samples to equilibrate the O<sub>2</sub> concentration with the atmospheric O<sub>2</sub> concentration.



- 2. Start the Labview program (shortcut 'oxdiff\_2')
- 3. Go to the warm-up menu, enter the ring numbers of your samples, and specify the path and filename. Press continue.



- 4. Calibration at the atmospheric (laboratory) O<sub>2</sub> concentration (around 20.9% at NTP)
  - a. Use the red lid as a fan to make air circulation around the chambers
  - b. When sensor readings are stable, press 'Accept 21%' to save the calibration values

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1	2	3	4	5	6	
24.0	-0.107	0.308	-0.493	0.359	0.0248	
7	8	9	10	11	12	
24.2	0.137	-0.217	0.0669	-0.032	0.8	

5. Remove the cloth and rubber ring from the samples and place them in the sample holders. Put both from below into the diffusion chamber and seal the chamber by turning the handle to the right.



- 6. Calibration at 0 % air:
  - a. Open for N<sub>2</sub> on the main valve (pressure ~1). Open the right valve on the chambers you are going to use.

- b. Open the inlet (left valve) and flush the chambers for 1 min until stability is reached (chamber values around 0.17-0.25%; may be changed in the 'settings' menu).
- c. Close the valves in the correct order (chamber by chamber), first the left valve, then the right valve and then quickly click 'Accept 0%'.
- 8. The measurement will start after accepting the 0% calibration data, and you will see an output of the oxygen concentration for each chamber in the plotting area.



9. The measurement will automatically stop after 120 minutes or after the O<sub>2</sub> concentration in each chamber has reached 21% (whichever occurs first; may be changed in the 'settings' menu).

### Calculations

The data from the diffusion measurement is transferred from the Lab-View system to an Excel file. Here the readings from the sensor (in voltage) are arranged in columns for each measurement. The first row with data in the spreadsheet is the calibration of the sensor in atmospheric air. The first row in the time series (t = 0) corresponds to the calibration of the sensor in an oxygen-free atmosphere, followed by the sensor readings at each measurement point ( $t \neq 0$ ). Data are converted to % O<sub>2</sub> in the following way

$$O_2 - \% = 20.9 \frac{(volt_t - volt_0)}{(volt_{atm} - volt_0)}$$

where *volt<sub>t</sub>* is the voltage at the different measurement points, *volt<sub>0</sub>* is the voltage in an oxygen-free atmosphere, and *volt<sub>atm</sub>* is the voltage in atmospheric air. 20.9 is the  $O_2$ -% in atmospheric air.

For each time step, calculate  $\ln\left(\frac{\Delta C_t}{\Delta C_0}\right)$ , and plot  $\ln\left(\frac{\Delta C_t}{\Delta C_0}\right)$  at the y-axis and time (t) at the x-axis. Use Eq.

[6] to calculate  $D_p$ .

### Reference

Rolston, D.E., and P. Moldrup. 2002. Gas diffusivity. pp. 1113-1139. *In* J.H. Dane 5 and G.C. Topp (ed.) Methods of soil analysis. Part 4. SSSA Book Ser. 5. SSSA, 6 Madison, WI.

# 4. Air-connected pore volume

#### Background

A pycnometer measures the volumes of irregularly shaped objects. The principle of the pycnometer is based on Boyle's law: A given amount of gas (moles) at a given temperature is the product of the volume and pressure of the gas. The background to this law is the thermal state equation for ideal gasses:

$$p V = n R T$$
<sup>[1]</sup>

Where *p* is the absolute pressure, *V* is the volume, *n* is the number of moles of gas, *R* is the gas constant, and *T* is the absolute temperature. If a constant mass of gas (n = constant) at a constant temperature *T* is distributed in two different volumes ( $V_1$  and  $V_2$ ) it will exert the pressures  $P_1$  and  $P_2$  in the two situations:

$$\left. \begin{array}{c} P_1 V_1 = n R T \\ and \\ P_2 V_2 = n R T \end{array} \right\} \quad \Rightarrow \quad P_1 V_1 = P_2 V_2 \qquad [2]$$

Used on soils, a pycnometer measures the volume of all the solid particles, water and entrapped air bubbles. If the outer dimensions of a sample are known, the measured volume can be subtracted from the former, and the remaining volume is the accessible (effective) air-filled pore volume.



Figure 1. Principle sketch of a pycnometer

The pycnometer is operated as follows:

The outlet valves (Fig. 1, valves 2 and 4) of the reservoir are closed, and with the help of a pump, either an overpressure or vacuum in the chamber is created. At the desired pressure, the inlet valve is closed (Fig. 1, valve 1), and the exact pressure is measured. Following this, the outlet valve of the sample chamber is closed (Figure 1, valve 3), the valve between the chambers is opened (Figure 1, valve 2), and a new, second pressure is recorded. With the knowledge of the two pressures and the volumes of the two chambers, it is possible to calculate the sample volume (Eq. [2]).

### Principle of the measurement of effective air-filled pore volume

Figure 2 shows the sketch of the pycnometer again, indicating where the different pressures arise.



Figure 2. Sketch of pycnometer showing the different pressure areas.

 $P_R$  is the pressure in the reservoir,  $P_C$  is the ambient pressure in the sample chamber, and  $P_{RC}$  is the resulting pressure in both chambers.  $V_R$  and  $V_C$  are the two involved volumes. Boyles's law is written with the above-used terms.

$$P_{\rm R} V_{\rm R} + P_{\rm C} V_{\rm C} = P_{\rm RC} \left( V_{\rm R} + V_{\rm C} \right)$$
[3]

Eq. [3] can be rearranged into Eq. [4] and [5]

$$P_{C} V_{C} - P_{RC} V_{C} = P_{RC} V_{R} - P_{R} V_{R}$$
[4]

$$V_{C} = V_{R} \frac{P_{RC} - P_{R}}{P_{C} - P_{RC}}$$
[5]

From Eq. [5], it is obvious that only pressure differences are involved. This allows working with relative (gauge) pressures (p) instead of absolute pressures (P). In this case, the ambient pressure ( $P_C$ ) disappears, and Eq. [5] can be written as:

$$V_C = V_R \frac{p_{RC} - p_R}{-p_{RC}}$$
[6]

With this general equation for calculating a volume based on pressures relative to the atmospheric pressure, it is possible to derive an equation for calculating the accessible pore volume of a soil sample if the outer volume of the sample is known. Figure 3 shows the same sketch for the pycnometer, showing the different pressures and volumes involved in calculating the accessible, air-filled pore volume. Here the initial pressure relative to the atmosphere is called p<sub>1</sub>, and the resulting relative pressure p<sub>2</sub>.



Figure 3. The different pressure and volumes involved in the calculation of the accessible, air-filled pores volume.

Eq. [7] and [8] show the final calculation of the accessible, effective air-filled pores volume ( $\varepsilon_{eff}$ ).

$$p_{1} V_{R} = p_{2} (V_{R} + V_{C} - V_{S} + \varepsilon_{eff})$$

$$\varepsilon_{eff} = \frac{p_{1} V_{R} - p_{2} (V_{R} + V_{C} - V_{S})}{p_{2}}$$
[8]

where  $V_R$  is the reservoir volume,  $V_S$  is the outer volume of the sample,  $V_C$  is the chamber volume,  $p_1$  is the original relative pressure, and  $p_2$  is the resulting relative pressure. If the chambers' volumes, the samples' dimensions and the two pressures are known, it is simple to calculate the accessible, effective air-filled pore volume of a given soil sample using Eq. [8].

### How to do:

#### Calibration

- 1) Before measuring any samples, you should always start by measuring a standard. If there are any equipment problems, a standard measurement will reveal this. The standards are plastic or metal cylinders with a known volume.
- 2) You should calibrate the instrument if the measurement deviates too much from the true value. Press the calibration- tab and follow the instructions.
- 3) Your measurements can be seen by pressing the measurements tab.

#### Measurements

Only the two lowest chambers (#1 and #2) are in use at the time being.

- 4) Open the chamber(s) and put in your sample(s).
- 5) Close the chamber. Make sure that all edges are clean. Put on the lid and turn the handle until the screw meets the stopper.

- 6) Choose a file to save your data in.
- 7) Write your sample numbers and remarks into the designated areas of the software
- 8) Press start
- 9) The program will now measure as many times on your sample as you have defined. The number of replicates is typically set to 5. The program will continue to measure until the deviation of all samples is under the defined deviation.

The deviation is calculated as (measurement-mean)/mean\*100.

10) While the program is running, be aware of any warning signals from the program. Constant beeping can be a sign of leaks in the equipment, often occurring at the lid. If so, a warning will be shown at the bottom of the program screen (Figure 3). In case of leaks, tighten the rubber in the lid with silicone grease.



Figure 1: The experimental set-up.

# Laboratory and Field Exercises: Merging Measurement and Modelling of Soil Physics



Figure 2. The program and its features.

# Laboratory and Field Exercises: Merging Measurement and Modelling of Soil Physics



Figure 3: The program and its features

# 5. Air permeability

### **Definition of air permeability**

Mass flow (also referred to as convective flow) is the movement of a fluid in response to a pressure gradient. The ability of a porous material (including soil) to conduct a fluid by this process is termed intrinsic permeability, which in theory, is independent of the flowing fluid. The air permeability ( $k_a$ ), thus, is an estimate of the intrinsic permeability when using air as the flowing fluid. Here, the intrinsic permeability is generally referred to simply as the permeability.

Air permeability is useful in the characterization of soil pores. Knowledge of  $k_a$  and its variation with soilwater content is also necessary for modelling convective air and gas transport in soil, for example, in relation to analyzing and optimizing soil vapour extraction systems for the clean-up of soils contaminated with volatile organic compounds.

At low pressure gradients, air flow through porous media is comparable to water flow. Fluid independent permeability (k) of the soil is most often estimated via Darcy's law from measurements of air or water flow, according to

$$q = \left(\frac{k}{\eta}\right) \left(\frac{\Delta p}{\Delta x}\right) \tag{1}$$

where q is flux density,  $\eta$  is the dynamic viscosity, p is pressure, and x is the distance in the flow direction.

Air permeability in the laboratory is calculated using an integration of Eq. (1),

$$Q = \frac{k_a \Delta p a_s}{\eta L_s} \tag{2}$$

where Q is the volumetric flow rate,  $\Delta p$  is the pressure difference across the sample,  $a_s$  is the cross-sectional area, and  $L_s$  is the length of the sample.

### Laboratory exercise

1. Place the sample on the steal sieve



2. Place the air permeability measuring chamber over the sample and press the red switch to inflate the air sealing around the sample.



3. Adjust the pressure to approximately -5 cm using the pressure controller



+



4. Read the flow from one of the three flow meters. Makes sure that air only flows through one of the flow meters.

If the left flow meter has to be used, use the conversion curve on page 3



5. If the flow is too low to be read from the mechanic flow meter, use the electronic flow meter.



6. Calculate the air permeability using Eq. [2]. Use the following constants:  $\Delta p = \Delta h \text{ g } \rho_w$ 

$$g = 9.816 \text{ m s}^{-2}$$

$$\label{eq:rhow} \begin{split} \rho_w &= 998.2 \ \text{kg} \ \text{m}^{\text{-3}} \\ \eta_{air} &= 1.827 \ 10^{\text{-5}} \ \text{kg} \ \text{m}^{\text{-1}} \ \text{s}^{\text{-1}} \end{split}$$



# 6. Saturated hydraulic conductivity in the lab (constant head)

# Background

The movement of water governs to a large extent the transport and fate of water, nutrients, and chemicals in the soil system. The soil properties that determine the behavior of the water movement are the water retention characteristic and the hydraulic conductivity. The water retention characteristic is the ability of the soil to store water at different soil matric potentials, whereas the hydraulic conductivity is defined as the ability of the soil to transmit water. The saturated hydraulic conductivity is the ability of the soil to transmit water apositive pressure head when the soil is fully saturated, i.e. when water is flowing in all pores of the soil.

At low pressure gradients the flow of water obeys Darcy's law

$$q = -K_s \frac{\Delta H}{L_s} \tag{1}$$

where *q* is the flux density  $[LT^{-1}]$ ,  $\Delta H$  is the hydraulic head [L] (see Figure), and  $L_s$  is the length of the soil sample [L]. As discovered from the equation, the flux density is proportional to the pressure head gradient  $(\Delta H/L_s)$  where the proportional factor  $K_s$   $[LT^{-1}]$  is termed the saturated hydraulic conductivity  $[LT^{-1}]$ . Darcy's law is valid for both saturated and unsaturated flows of water.

# Principle

A soil column is placed in a container on a wire mesh allowing water to flow through the column from the bottom to the top. A removable top is mounted on the column and sealed with inflatable air seals. This top is connected to the outside of the container by a tube. The soil in the column is slowly wetted over night by raising the water until it leaves the outlet: B. As the inner water level can't go higher than the top of the outlet A, a hydraulic head ( $\Delta$ H) will arise and water will infiltrate through the column. The infiltration water is collected through outlet A and measured during time on a balance.



When equilibrium arises, discharge rate (*Q*)  $[L^{3}T^{-1}]$  and hydraulic head ( $\Delta H$ ) is recorded and K<sub>s</sub> can be calculated using eq. [1].

# Procedure



When you do this analysis, you become extremely happy :-)

## Saturation:

Open the orange valve and start inline pump (Fig. 1) and create a  $\Delta H$  in the top container at 10 cm.

Stop the inline pump and close the valve.



Figure 1 Inline pump and valve

Place the sample on the wire mesh in the container. The sample have to be put down obliquely in order to prevent air being trapped at the bottom of the soil sample (see sketch below):





Drain water from the tank by removing the thick gray pipe (Fig. 2) and open the orange valve again. Stop the drainage when the water level is 3-5 cm over the bottom of the tank.

Figure 2. Gray pipe

Put on the black top (Fig. 3) and connect the white tube (Fig. 2). This will inflate the orange air seal inside the top (Figs. 4 and 5).



Figure 3: The equipment



Figure 4: Deflated air seal



Figure 5: Inflated air seal



Figure 6: 3-way valves

Connect to thick transparent tubes (Fig. 3) to the outlet from the container.



pump

On the containers on the balance, check that the lower transparent plastic pipe is open. The black rubber tube inside the transparent pipe should be deflated. If not, open it by turning the three way valves, which are placed on the end of the equipment (Fig. 6).

Start the peristaltic pump by moving the switch to the upward position (Fig. 7).

### Measurement:

Open valve and start inline pump.



Figure 8: Metal pipe

While the water level is raising check which  $\Delta H$  gives a suitable flow. By measuring the difference in water levels inside and outside the black top  $\Delta H$  can be determined. The flow should not be larger than 500 l/min. If the flow is too high new pathways through the sample might be created especially if the stability of the soil structure in the sample is pure. If the flow is too high, attach a metal tube in the hole inside the container. Find a tube with a suitable length. With very low flow use the shortest metal tube.

When the outer water level has reached the outlet of the thick gray pipe and the inner water level reaches the metal pipe, the measurements can begin. On the container on the balance, close the lowest transparent pipe by turning the three-way valve at the end of the equipment.

Start logging from the computer. Measure the pressure head.

If the volume of water in the container on the balance exceeds the maximum of the balance (that's when no data are collected on the screen) remove the water in the container. This can be done by opening the lowest transparent pipe by turning the three-way valve at the end of the equipment and close again when empty.

When the flow has been constant for around 15 minutes stop the logging and press 'save'. A new form containing the measured data will appear.

Remove data from non-steady periods by marking the unwanted rows and by pressing 'ctrl k.'.

Then press 'save' again (remember to check if the file paths are correct and change them if necessary)

The data are saved in two files:

A file with all the data

A file with average flow, diameter and height of ring, pressure head, and the K<sub>sat</sub> value only.

## Removing samples after analysis:

Empty the upper tank by opening the red valve at the end of the container and removing the gray thick pipe inside the tank.

Deflate the orange air seal by removing the white tubes connected to the orange air seals. Take care of your ears. The sound is quite high.

Open the lowest transparent pipe by turning the three-way valve and the end of the equipment.

When the water has left the container, leave the samples dripping for some time before removing them.

# 7. Saturated hydraulic conductivity in the field (SATURO)

# Background

Soil saturated hydraulic conductivity (K<sub>s</sub>) is defined as the capability of the soil in transporting water under saturated conditions (e.g., after heavy rainfall or irrigation). K<sub>s</sub> is a very important parameter in soil water and solute transport models. Soil texture strongly affects the soil K<sub>s</sub>. For example, sandy soil, due to its larger pore space, has higher K<sub>s</sub> than clay soil. Average K<sub>s</sub> values for different soil textures are shown in the table below (Hydrus-1D software, version 4.17).

Soil texture	K <sub>s</sub> (cm d <sup>-1</sup> )
Sand	712.8
Loamy sand	350.2
Sandy loam	106.1
Loam	24.9
Silt	6
Silt loam	10.8
Sandy clay loam	31.4
Clay loam	6.24
Silty clay loam	1.68
Sandy clay	2.88
Silty clay	0.48
Clay	4.8

Soil saturated hydraulic conductivity (Ks) across the United States Department of Agriculture (USDA) textural classes

# Device

SATURO is an automated device designed for measuring soil  $K_s$  in the field. It applies a multi pressure head analysis method for 3D flow corrections from a single-ring infiltrometer. It allows for quick measurements of  $K_s$  that need no postprocessing of the data. SATURO reduces errors in the measurement of  $K_s$  compared to laboratory measurements. The figure below shows an installed SATURO device.



Graphical representation of the installed SATURO device

# Setup of the device

The steps below should be followed to successfully set up and run the SATURO device:

- Select the desired measurement location in the field and remove rocks and other large debris from the surface where the ring will be installed.
- Place the ring on the soil, cap it with the plate, and hammer on the inner circle of the plate until the ring is levelled with the top of the soil, ensuring the lack of gaps between the soil and the ringside walls. Then, remove the plate.

**Important point:** there are two types of rings in two depths (5 cm and 10 cm). The 5-cm ring is used for sites with good soil structure. The 5-cm ring is generally recommended for most sites. The 10-cm ring is used for sites with a loose soil surface or sites with high fluxes because of the existence of macropores.

- Connect the infiltrometer head to the ring and connect the tubes and device cable to the right fittings.
- Put the water tube in a water source (e.g., in a large water container filled with water). To ensure that the tube remains underwater, place it at the bottom of the container. A tape can be used to fix the tube and prevent it from being removed.
- Place the SATURO device on a stable surface and turn it on.
- Select a name for the measurement, configure settings according to the soil conditions, and start the measurement.

The table below indicates the appropriate configurations for different soil types.

Soil Type	Soak Time (min)	Low Pressure Head (cm)	High Pressure Head (cm)	Hold Time at Pressure (min)	Pressure Cycles (count)	Total Run Time (min)
Dry loamy sand	25	5	10	15	3	115
Wet loamy sand	15	5	10	15	2	75
Dry silt loam	30	5	15	20	3	150
Wet silt loam	15	5	15	20	2	95
Dry clay (poor structure)	30	5	20	25	3	180
Wet clay (poor structure)	15	5	20	25	2	115
Dry clay (strong structure)	25	5	10	20	3	145

## SATURO device configurations for different soil types.

NOTE: These values are a rough starting point only. Soil conditions dictate the optimal settings for the test. Use lower pressure head settings for soils dominated by macropore flow. If necessary, reduce the pressure head settings to allow the instrument to keep up with the flow rates.

# 8. Unsaturated hydraulic conductivity in the lab (drip infiltrometer)

# Background

Most of the water flow in the soil occurs at unsaturated conditions. At this state, water flow is not only driven by gravity but also due to a gradient in the matric pressure head. The relation between the unsaturated hydraulic conductivity (k) and the matric pressure head (h) is highly dynamic and even small changes in h may cause a dramatic change in k (Fig. 1).



Figure 1. Examples of the relation between k and h (suction head/matric pressure head) measured on two different soils.

# Set-up of instrument

We measure the unsaturated hydraulic conductivity (k(h)) in this exercise by means of a drip infiltrometer.



Figure 2. Schematic of the drip infiltrometer

Soil column: Undisturbed soil column 20 cm in height and 20 cm in diameter.

Irrigation head: Artificial rainwater (0.01 M CaCl2) is added from the top with a rotational irrigation head.

Ceramic plate: Controls the suction at the bottom of the soil column.

<u>Tensiometers</u>: Five tensiometers measure the matric potential at different positions in the soil column.

Pressure transducers: Readings from the pressure transducers are sent to a PC controlling the analysis.

<u>Vacuum container</u>: Controls the suction at the ceramic plate and collects the infiltrating water from the ceramic plate.

Balance: Measures the inflow through the irrigation head in time. Data is send to the PC.

1. When starting the analysis, the pump rate and vacuum is chosen by the user. The starting pump ratio is set in order to reach a matric potential as close to zero as possible.

2. Suction at a ceramic plate is adjusted until tensiometers show almost similar readings (gravity flow only).

3. The PC controls for 1 hour that steady state conditions exist

4. Readings are performed for the flux, q, and matric potential, h.

5. The unsaturated hydraulic conductivity, k(h), at the specific value of h is calculated based on Darcy's law:

$$q = -k\frac{dH}{dz} = -k\frac{d(h+z)}{dz} = -k\frac{dh+dz}{dz} = -k\left(\frac{dh}{dz}+1\right)$$

where q = water flux [cm/d], k = conductivity [cm/d], H = hydraulic head [cm], z = gravitational head or height above a reference level [cm], h = matric pressure head [cm], and H = h+z.

6. The pump rate is lowered with approximately 20% and the measurement procedure is repeated at a lower matric potential. Analysis ends at the lowest possible value of q.

The control of the measurements is fully automatic. The program finds the optimal settings independent on the user settings. The settings and the status of the measurement procedure is be controlled by a labVIEW user interface.

### Procedure

The soil sample is before the start of the exercise slowly saturated in the drainage box during a period of one day (depending on the soil type). After that, the sample is drained to a matric pressure head of -30 cm H2O relative to the centre of the column.

The soil sample is carefully removed from the drainage box and placed on the table.

Drill the holes for the tensiometers into the sample using the auger set.

Carefully place the soil sample on the ceramic plate

De-air the tensiometers.

Insert the tensiometer into the soil sample in the right order. Turn the small "cups "upward and be sure that the tensiometers are turned off.

Fill up the small cups with water. The water level in the small cups is 3 cm above the point of measurement.

Use duct tape to glue on top ring on the sample. Place raid head on the top ring.

Select appropriate vacuum and pump rate

Start up the drip infiltrometer

Follow the instructions from the program.

Wait until the next day for the measurement to be finished.

Use Darcys law to calculate the hydraulic conductivity for the given matric pressure head. Make a linear regression of the matric pressure head for the three middle tensiometers. Calculate h and k for the centre of the soil sample.



Figure 3. Example of pressure heads measured in a soil sample at one specific step in the analysis.



Figure 4. Example of the unsaturated hydraulic conductivity for a sandy topsoil measured at several matric pressure heads.

# 9. Unsaturated hydraulic conductivity in the field (tension infiltrometer)

### Background

Tension infiltrometers are designed to measure the unsaturated hydraulic properties of soils. Water is allowed to infiltrate into the soil at a rate which is slower than when water is ponded on the soil surface. This is accomplished by maintaining a small negative pressure on the water as it is infiltrating into the soil. By applying a small negative pressure (or tension) on the water as it is infiltrating, water will not enter the large cracks or wormholes, but infiltrate into the soil matrix.

## Underlying theoretical principles

Hydraulic conductivity k(h) as a function water tension h in soils near saturation can be described according to Gardner (1958)

$$k(h) = K_{sat} \exp(\alpha h)$$
[2]

where K<sub>sat</sub> is the saturated hydraulic conductivity and h is the soil moisture potential (here in positive values).

According to Wooding (1968), the following applies to the steady-state flow Q (volume/time) from a circular infiltration area (radius a) into the infinite soil:

$$Q = \pi a^2 k(h) \left( 1 + \frac{4}{\pi \alpha a} \right)$$
[3]

From experimental determination of  $K_{sat}$  and  $\alpha$ , the infiltration test can be made with different water tensions (soil moisture potentials).

### Methodology of test procedure

Infiltration tests can be made at any water tension in the near-saturated range. For neighboring values of the chosen water tensions  $(h_1, h_2)$ :

$$\frac{Q_1}{\pi a^2} = K_{sat} \exp\left(\alpha h_1\right) \left(1 + \frac{4}{\pi \alpha a}\right)$$
[4]

$$\frac{Q_2}{\pi a^2} = K_{sat} \exp\left(\alpha h_2\right) \left(1 + \frac{4}{\pi \alpha a}\right)$$
[5]

By way of division we get:

$$\alpha = \frac{\ln\left(\frac{Q_1}{Q_2}\right)}{h_1 - h_2} (h_1, h_2 < 0)$$
[6]

### and for the hydraulic conductivities we get:

$$k(h_1) = \frac{\frac{Q_1}{\pi a^2}}{\left(1 + \frac{4}{\pi \alpha a}\right)}$$
[7]





Fig. 1. Schematic of the infiltrometer. The effective pressure head on the soil surface can be determined with a precision of 1 mm from the difference of the height of the water level in the standpipe and the negative pressure head at the U-tube manometer. The zero point of the scale of the standpipe is at the soil surface  $(U_s = negative \text{ pressure at the U-tube manometer}, H = height of the water table in the standpipe, H_K = infiltration chamber height, and T = submergence depth of the air pipe).$ 

The effective water tension (h) on the soil surface can be chosen via the depth of submergence [T] (Fig. 1):

$$h = H - Us \quad (h < 0) \tag{8}$$

For the exercise you measure the hydraulic conductivity at two different matric heads (-5 and -10 cm).

# 10. Column leaching

# Background

Leaching experiments provide information about the ability of a soil to infiltrate water and solutes which play an important role in crop production, risk of surface runoff, recharge of groundwater reservoirs, groundwater pollution, etc. By analyzing solute tracer movement we can determine flow parameters and structural properties of the soil core. Measuring the particle losses we can assess the leaching risk of colloid and colloidbound contaminants.

In this leaching exercise we are irrigating different soil cores ( $\emptyset = 20$  cm, h= 20 cm) with artificial rainwater and tritium is applied as an inert tracer. Several measurements are carried out on the column effluent (incl. the tracer activity of tritium).



Figure 1. Sketch and picture of the leaching set-up.

# Before the leaching experiment (setting the initial conditions):

 Prepare the artificial soil-water solution (0.652 mM NaCl, 0.025 mM KCl, 1.842 mM CaCl<sub>2</sub> and 0.255 mM MgCl<sub>2</sub>; <u>pH = 6.28; EC = 0.65 mmho</u>) [To prepare the soilwater dissolve 3.18g NaCl, 0.19g KCl, 27.08g CaCl<sub>2</sub>·2H<sub>2</sub>O and 5.18g MgCl<sub>2</sub>·6H<sub>2</sub>O in 100 L water].



Figure 2: Preparation of soil-water solution.

- Saturate the soil sample for about 3 days in the artificial soil-water solution in a sand box
- Drain the soil to -20 cm matric potential relative to the mid-section of the column for about 3 days



Figure 3: Concept of the sand boxes for column leaching and drainage.

• Weigh the soil after 3 days of drainage

# Initiating the leaching experiment:

 Prepare the artificial rainwater solution (0.012 mM CaCl<sub>2</sub>, 0.015 mM MgCl<sub>2</sub> and 0.121 mM NaCl; <u>EC = 0.028 mmho; pH = 5.68</u>) [To prepare the rainwater add 0.707 g NaCl, 0.176 g CaCl<sub>2</sub>.2H<sub>2</sub>O and 0.305 g of MgCl<sub>2</sub>.6H<sub>2</sub>O in 100 L water].

The set-up for irrigation consists of a tank with artificial rainwater, a pump and an irrigation head with 44 needles.



Figure 4: The rain/irrigation head with rain water coming out of the 44 needles

• The columns are irrigated with 10 mm artificial rain water per hour. Make sure that the intensity of rainwater is 10 mm h<sup>-1</sup> by calibrating the pumps. To check the irrigation intensity, first adjust the

settings of the pump to obtain a discharge of 10 mm  $h^{-1}$  from the pump, start the pump for irrigation and measure the rainfall intensity by measuring the amount of water collected per unit time from the irrigation set-up. You may have to replace some needles if they are blocked and also adjust the settings in the pump so the outflow is 10 mm  $h^{-1}$ . Follow a similar procedure for the tritium pump.

• Place the soil column on a steel grid with a mesh size of 1 mm in the leaching experiment set-up. Lower boundary is free drainage in order to promote macropore flow.



Figure 5. Soil column ready in the leaching set-up

• Effluent is collected through a funnel leading down to a number of weighted plastic bottles placed on a rotating auto sampler. The empty plastic bottles have to be weighed before the start of the experiment. Furthermore, the bottles have to be numbered according to their location in the roundabout. Weight and number are noted in the excel-sheet in the laboratory computer.



Figure 6. The weighed and numbered bottles in the automated leaching roundabout.

- Start the pump and apply the artificial rainwater at an intensity of 10 mm h<sup>-1</sup>. Record the time as start of irrigation.
- The time taken for the first drop of water to exit the column is recorded and defined as water breakthrough time. Water breakthrough time is recorded, and from water breakthrough time the automatic roundabout is started and collects effluent from the columns automatically in sample intervals of 6x10 min, 12x15 min and 6x30 min.

- As the bottles fill up, they are weighed and when the outflow is steady tritium is applied (<u>time =0</u>) as a pulse for 10 minutes. Tritium is a radioactive conservative tracer and is applied with the same intensity as the rain water.
- Continuously, each bottle is weighted, and analyzed for pH, electric conductivity (EC), turbidity (NTU), tritium activity (first from the bottle where tritium was applied), and particulate phosphorus concentration (calculated as the total phosphorus concentration minus the dissolved phosphorus concentration).
- For tritium activity (DPM): 1 ml of effluent + 2 ml elga water and 17 ml scintillation fluid. DPM: Disintegrations per minute, the SI unit of DPM is Becquerels (Bq) Bq: Disintegrations per second; 1 Bq = 60 DPM
- Measure the total effluent drained after the irrigation has stopped and also the weight of the soil column.

You will be provided with data sheets with all of the above mentioned measurements for columns from Jyndevad and Estrup. The sample intervals in these sheets will vary, also from the intervals given above, depending on the column.

# 11. Assignments

# Soil-water retention, gas, and water transport (100 cm<sup>3</sup> soil samples)

Calculate the following from the measurements in the lab:

- 1. Construct the water retention curve for the two soil types. Make a plot with volumetric water content on the x-axis and matric potential on the y-axis.
- 2. Predict five additional points using the Karup model which was presented for you Monday
- 3. Fit the water retention curve with the Van-Genuchten equation (use RETC or Excel)
- 4. Calculate the air-filled porosity,  $\varepsilon$ , at the two matric potentials at -30 and -100 cm
- 5. Calculate the air-connected porosity,  $\varepsilon_{ac}$  (from pycnometer)
- 6. Calculate the diffusion coefficient, D<sub>p</sub>
- 7. Calculate the relative diffusivity,  $D_p/D_o$ , where  $D_o$  is equal to 0.2 cm<sup>2</sup> s<sup>-1</sup>
- 8. Calculate the apparent tortuosity,  $T_{app} = (\epsilon/(D_p/D_o))^{1/2}$
- 9. Calculate the tortuosity,  $T = (\epsilon_{ac}/(D_p/D_o))^{\frac{1}{2}}$
- 10. Calculate the length of the diffusion pathway,  $L = T L_{column}$
- 11. Calculate the air permeability, ka
- 12. Calculate the equivalent pore diameter,  $D_{eq} = (8k_a/(D_p/D_o))^{\frac{1}{2}}$
- 13. Suggest a best prediction model for  $D_p/D_o$
- 14. Suggest a best prediction model for  $k_a$

# Water transport in the lab and in the field (Ks and k(h))

- 1. Calculate the saturated hydraulic conductivity (Ks) using Darcy's law
- Calculate the unsaturated hydraulic conductivity (k(h)) for the various matric potential using Darcy's law
- Make a plot of the hydraulic conductivity for all sites (lab and field) in the same figure. The conductivity on the y-axis and matric potential on the x-axis. Include both K<sub>s</sub> and k(h) in the graph.
- 4. Based on the results, reflect on the following

- a. How do the two soils differ in respect to their hydraulic properties?
- b. How will this be reflected in the possible differences in water transport?

# Column leaching (K<sub>s</sub> and k(h))

- 1. Plot outflow (ml/hour) as a function of time
  - Do you reach steady outflow?
  - Why do the outflow fluctuate more for some columns than for others?
  - Evaluate the water breakthrough times from the differently textured columns
- 2. Plot EC and pH as a function of outflow (in mm)
  - Comment on what you see in the graphs
  - Why is it important to use mm instead of time in the x-axis?
  - What can we use the curves of pH and EC for (hint: consider the EC in the soil water contra EC in the rain water)?
- 3. Plot particle concentration as a function of outflow

The measured NTU can be converted to particle concentration (mg  $L^{-1}$ ) using the following equation:

Concentration of particles 
$$\left(\frac{mg}{L}\right) = 3.14 \cdot NTU^{0.75}$$

- Explain the particle concentration curves for the three locations, how do they differ and why?
- 4. Plot particulate phosphorus concentration as a function of outflow and comment on the graphs
- 5. Plot particulate phosphorus concentration as a function of particle concentration in each bottle (in an xy-plot with correlation for each location) and comment on what you see
  - Which consequences does this relation have for the environment (e.g., after tillage operations)?
- 6. Plot the relative tritium activity as a function of time in order to obtain the breakthrough curves. For the tritium breakthrough curves time = 0 min is when tritium is applied (not when the irrigation started initially). In the sheets given to you there is a column called "Accumulated tritium time" which should be used for plotting the breakthrough curves.
  - Are the curves perfectly bell-shaped or right- or left skewed? What can the shape tell you about the transport phenomena in the three soil columns?
  - How can their form be explained by the different soil textures?