# ecoTech Automated Column Percolation System

Manual

*July 2019, Subject to modifications!*

# Manual ecoTech Automated Column Percolation System

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# 1. Safety Precautions

Before using the system for the first time, be sure to read this operating manual and note the following:

- The system is operated with 230 V DC mains voltage! Make sure that the connection between the system and the 230 V mains cable is earthed!
- The mains plug must be removed before opening the control cabinet!
- The system must not be operated in ex-protected rooms or in the presence of explosive or flammable media!
- It is essential to ensure the stability of the entire system! Possibly, additional measures must be taken (anchoring the shelf in the floor or on the wall, etc.)!
- The system is only intended for carrying out column percolation tests!
- Repairs may only be carried out by a specialist and after consultation with ecoTech GmbH Umwelt-Messsysteme! Unauthorized interventions are at your own risk and lead to the loss of warranty claims!
- After filling the column, all components of the column head and base and the outside of the column must be clean, dry and free of grease! Otherwise, leaks can endanger proper operation.
- After filling the column, check it for stability!
- Before every operation of the system, all hoses and hose connections for damage, leaks, correct stop and closure must be checked and, if necessary, replaced!
- Prior to each percolation, all incoming hoses to the saturation sensors must be free from residue and eluate. The LEDs of the saturation sensors light up green at this time!
- The company ecoTech Umwelt-Meßsysteme GmbH is not liable for damage caused by the operation of the system!
- We assume that the GLP guidelines are adhered to.

## 2. General set-up

The system has been designed as open rack system, to ensure easy access to all parts of the system. Its basic version is equipped with one column, the system is expandable with up to four columns. In the following, the extended version with four columns is taken as basis for this manual. All functions between the different versions are identical, but the number of columns.

*Figure 1: Installation with four columns housed in its three part rack system (center rack and two end racks) with its individual modules (1: eluent reservoir; 2: pump; 3: leaching columns; 4: hydraulic distributor; 5: sampling bottles)*

The following figure illustrates the process sequence during a percolation (Figure 2):

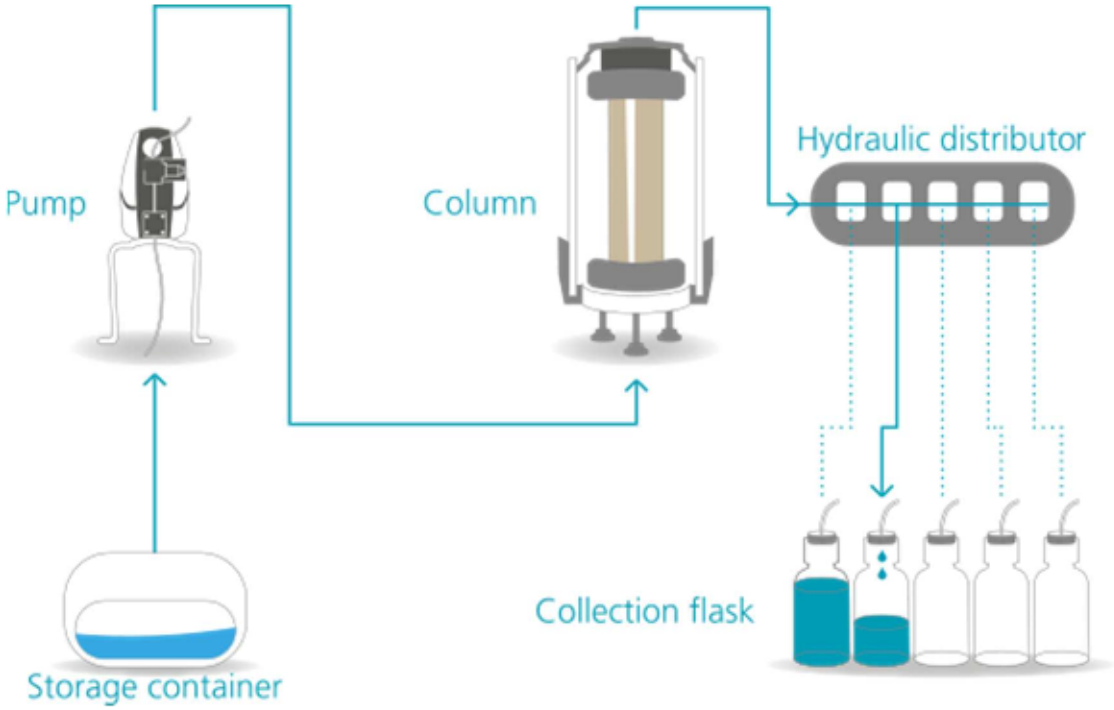



*Figure 2: Main components and process sequence during a percolation. Arrows indicate flow direction of liquid.*

The eluent is pumped from a storage tank by means of a pump from the bottom up through the leaching column. After leaving the leaching column, the leachate is distributed by the hydraulic distributor in the absence of air into the different fractions, i.e. different sampling bottles.

The automated percolation process is controlled by a programmable logic controller (PLC), housed in an electrical cabinet. The electrical cabinet contains, besides the PLC, all control devices, including the interface for the hydraulic valves, pumps, and sensors. This whole unit is herein after referred to as control unit. The control unit is connected to a PC workstation (either directly or via a local network) and receives the necessary process data (flow rate and fraction switching time) from there via the PC software AIO-Control7 for the realization of a percolation test.

Several control units, hence several leaching columns, can be operated from one central PC workstation. The actual status of the running percolation can be checked from further PC workstations within the same local network.

# 3. Test methods

All following mentioned test methods are up-flow percolation tests under saturated conditions.

## 3.1. Basic characterization according to DIN 19528

The basic characterization test according to the German DIN 19528 is used to gain information about the short- and long-term leaching behavior of solid material. This test involves four different fractions up to a L/S (liquid/solid) ratio of 4,0 (Table 1). The column has to be completely saturated within 2 h. The contact time between the eluent and the test material during the percolation is set at 5 hours according to norm, the system adjusts automatically the flow rate for each test material conforming to this standard.

*Table 1: Stipulated L/S ratios for the basis characterisation test according to DIN 19528 (MD = dry mass of test material)*

| Fraction | L/S ratio ± tolerance | Expected eluate volume |
|---|---|---|
| 1 | 0,3 ± 0,05 | (0,3 ± 0,05) x MD |
| 2 | 1,0 ± 0,2 | (0,7 ± 0,2) x MD |
| 3 | 2,0 ± 0,4 | (1,0 ± 0,4) x MD |
| 4 | 4,0 ± 0,8 | (2,0 ± 0,8) x MD |

## 3.2. Compliance test according to DIN 19528

The compliance test is used to test whether the test material meets a specific behavior or specific reference values. It is used to check the results of previous basic characterization tests during routine validations tests.

While maintaining the same contact time between sample material and eluent (5 hours) like in the previous basic characterization test, all leachate is collected in one sampling bottle until a L/S ratio of 2,0.

## 3.3. Blank value test according to DIN 19528

All system components should be subjected routinely to a blank value test. A leaching column just filled with quantity of quartz sand normally used for the filter bed during the tests is subjected to a leaching until a L/S ratio = 2. Basically, this test is a compliance test like in chapter 3.2 , but without any test material.

## 3.4. NEN 7373

This Dutch standard describes a column test to determine the availability of inorganic components for leaching from solid earthy and stony materials.

The column has to be completely saturated within 6 (± 1) h, afterwards the circulation stops for a period of 18 h to 72 h. This waiting time is intended to achieve a chemical equilibrium between the solid matter and the liquid. After that, the test starts collecting seven different fractions:

*Table 2: Stipulated L/S ratios for NEN7373 (MD = dry mass of test material)*

| Fraction | L/S ratio ± tolerance | Expected eluate volume |
|---|---|---|
| 1 | 0,1 ± 0,01 | (0,1 ± 0,01) x MD |
| 2 | 0,2 ± 0,02 | (0,1 ± 0,01) x MD |
| 3 | 0,5 ± 0,05 | (0,3 ± 0,03) x MD |
| 4 | 1,0 ± 0,1 | (0,5 ± 0,05) x MD |
| 5 | 2,0 ± 0,2 | (1,0 ± 0,1) x MD |
| 6 | 5,0 ± 0,5 | (3,0 ± 0,3) x MD |
| 7 | 10,0 ± 1,0 | (5,0 ± 0,5) x MD |

### 3.5. EN 14405

A flow rate of 15 cm/day is expected to saturate the column completely. Once the column has reached this state the circulation stops for a period of 16 h to 72 h. This waiting time is intended to achieve a chemical equilibrium between the solid matter and the liquid. After that, the test starts collecting seven different fractions:

*Table 3: Stipulated L/S ratios for EN14405 (MD = dry mass of test material)*

| Fraction | L/S ratio ± tolerance | Expected eluate volume |
|---|---|---|
| 1 | 0,1 ± 0,02 | (0,1 ± 0,02) x MD |
| 2 | 0,2 ± 0,04 | (0,1 ± 0,02) x MD |
| 3 | 0,5 ± 0,08 | (0,3 ± 0,05) x MD |
| 4 | 1,0 ± 0,15 | (0,5 ± 0,1) x MD |
| 5 | 2,0 ± 0,3 | (1,0 ± 0,2) x MD |
| 6 | 5,0 ± 0,4 | (3,0 ± 0,2) x MD |
| 7 | 10,0 ± 1,0 | (5,0 ± 0,2) x MD |

### 3.6. Custom percolation

Apart from the above-mentioned national standard tests, the system offers the possibility to define its own test procedure. Up to four fractions (additionally expandable to up to 7 fractions) can be collected. The volume per collected fraction is freely selectable. Additionally, it can be chosen between L/S ratio and PV as controlling variable. The flow rate during the saturation of the column and later test is also freely selectable. This program also gives the possibility to stop the percolation after the column is completely saturated. This waiting time is intended to achieve a chemical equilibrium between the solid matter and the liquid. After that, the test starts automatically.

Details on the data entry are given in chapter 4.2.7 starting on page 25.

# 4. System operation

## 4.1. Preparations

### 4.1.1. Packing and connection of leaching columns

Care must be taken that all elements of the column closures are clean, dry and free from any grease. This is important to ensure the leak tightness of the system. All threads must be clean of any residues before screwing, any remaining particles could damage the components and therefore disturb the whole function of the system.

Before filling the leaching columns with the test material, the inlet tube must be attached to the column. It has to be passed through the borehole in the including grey PVC tube fitting, fitted with a seal ring at the threaded end and finally screwed with the column base together.

Before closing the tube fitting tightly, care must be taken that the tube extends into the column interior space to reach into the later sand filter bed (~0,5 cm). The tube fitting must be closed hand-tightly.

To protect the tube from intrusion of filter sand some quartz wool is placed over the tube end.

In a next step, the glass tube with an O-ring seal is placed in the column base and the O-ring seal is pushed by hand into its basis.

After mounting the clamping ring the union nut (small columns) is screwed with the basis together and the clamp ring together with its fastener clamp (big column) are pressed against the basis respectively.

With the assembly of the large columns (435.100SC) care must be taken that the opposed fastener clamps are always closed simultaneously. One-sided closure can lead to a none equal contact pressure and disturb the leak tightness of the system.

The German DIN 19528 defines how to pack the leaching column with the test material. At first a 2 cm high filter sand bed is filled into the column, to ensure a regular flow of the eluent over the whole diameter of the column. The grain size of the filter sand should be 0,6-1,2 mm according to DIN 19528.

The leaching column including the lower sand filter has to be tared now.

After that the test material is filled in by layers (H = 5cm).

**Notice**

Own experience has shown that the quartz wool that covers the tube end slides easily when filling the column with the filter sand. To prevent it, the wool should be fixed with a thin bar until it is covered with the filter material.

It is also advisable to rotate the column axially during the filling process with the test material several times. Thus an unwanted separation of the material within the column is avoided.

This applies above all when adding 80 % quartz sand to the test materials with a low permeability, according to DIN 19528.

Layering within the test material might provoke preferential flow, an uneven saturation of the material and a different flow rate through the column than expected, in summary causing possible inaccurate or falsified test results.

After filling in each layer of test material into the leaching column, the material must be compacted. The DIN 19528 allows different methods: Drop weight, sieve shaker or by tapping the sides of the leaching column with a rubber mallet.

We recommend compacting the material with a drop weight to archive highest possible reproducibility. According to DIN 19528 – Annex E a drop weight should exert 1,5-2 g/cm² on the surface of the test material.

Supported by the standard specification FprCEN ISO/TS 21268-3:2009 (Soil quality – Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials – Part 3: Up-flow percolation test) a drop weight with a density of 6,4 g/cm³ can be used. For a leaching column diameter of 65 mm it corresponds with a mass of 212 g. This weight is lifted 200 mm guided by a leading rod and dropped from this height. This step has to be repeated twice. After filling the test material into the leaching column, the column has to be tared now a second time, to know the exact mass of sampling material inside the column.

An upper sand filter bed with a height of 20 ± 5 mm is introduced than into the column. The column has to be completely full now.

Before closing the column with the upper lid, the outlet tube has to be attached to the column lid. It has to be passed through the borehole in the including grey PVC tube fitting, fitted with a seal ring at the threaded end and finally screwed with the column base together. Before closing the tube fitting tightly care must be taken that the tube extends into the column interior space to reach into the later sand filter bed. The tube fitting is now closed hand-tight. To protect the tube from the intrusion of filter sand, some quartz wool is placed over the tube end. Care must be taken before closing the leaching columns; all components must be clean, dry and free from grease. Screw the union nut hand-tight (small columns), and watch an even clamping (big column), respectively.

If not enough test material is available, the lower sand filter bed can compensate the lack of material according to DIN 19528. To do so, the column must be packed upside down and then turned before connecting the tubes.

When the leaching column is filled, weighted, and closed, the corresponding tubes have to be connected with the hydraulic distributor (column outlet) and the tee connector (column inlet) (Figure 3).

*Figure 3: Tee connector between pump, pressure sensor and leaching column*

### 4.1.2. Sampling bottles

All sampling bottles have screw caps with multiple distributors (figure 13), one acting as inlet for the leachate: The tube coming from the hydraulic distributor has to be inserted into the opening and screwed to fix it. The second available opening acts as venting or as spillway to a second subsequent bottle.

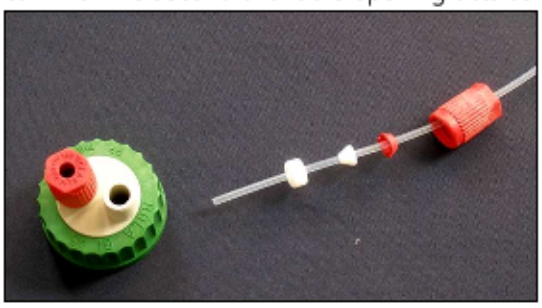


*Figure 4: Screw cap with multiple distributor and lab screw joints*

When using the expected leachate volume is larger than the volume of the sampling bottle, several bottles have to be connected to gain a higher total sampling volume:

A connecting tube has to be inserted into the first bottle until the calibration mark, marking the maximal filling level of this vessel. The other end of this tube has to be introduced into the subsequent bottle. When the leachate level in the first bottle reaches the calibration mark, it is pressed due to a slight overpressure in this bottle into the next bottle. The principle of communicating vessels makes sure that flow from one bottle to the other does not break.

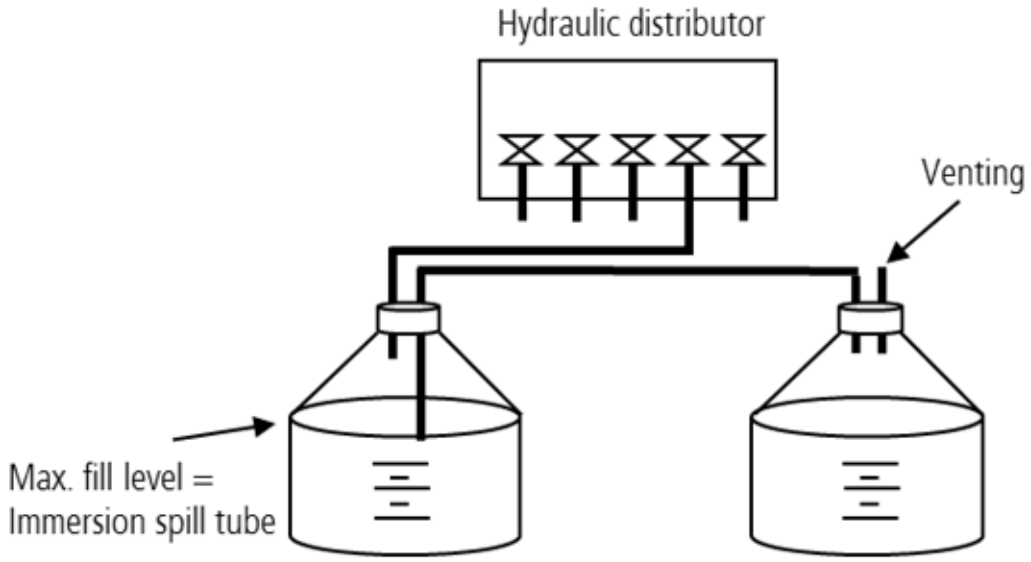



*Figure 5: Linking of several sampling bottles to distribute larger volumes of leachate*

A manual exchange of the sampling bottles is thus not necessary. Furthermore the maximum weight to be lifted for the user is reduced to the weight of one filled 5 L bottle.

Depending on the calculated leachate to be created, several sampling bottles have to be linked in series. Attention: Check before starting a new percolation that the sump capacity in each fraction is big enough to take up the calculated leachate volumes (figure 26 on p. 43). When several bottles are linked in series, the last one has a ventilation opening. To reduce this opening, a hollow needle or a piece of a tube can be introduced into this opening to minimize the contact with the ambient atmosphere.

To ensure the proper functioning of the system, it is important that all bottles are closed tightly and that all laboratory screw joints are screwed into the screw caps.

**Before starting a new test, make sure check whether the size of the connected sampling bottles corresponds to the expected leachate volume!**

### 4.1.3. Eluent reservoir

To the fill the eluent reservoir the ~10 cm wide inspection opening on the upper side of the container must be opened. Care must be taken, that neither pollution, nor objects fall into the vessel while open! They could later be aspirated by the conveying tube of the pump and seriously damage the system, or at least falsify the test results. To fill the container at an external DI water supply, the eluent reservoir is rollable and has a removable push rod.

To disconnect the reservoir from the system, the conveying tube of the pump must be disconnected. This happens by unscrewing the tap on the upper side of the container or by loosening the tube fitting and simply extracting the tube from the reservoir.

After filling and accurate closing of the inspection opening, the conveying tube has to be passed through its corresponding inlet/outlet hole and locked. Please make sure that the tube is inserted deep enough to have contact with the bottom of the container, to use the whole volume of the eluent reservoir.

A hole in one of the upper corners of the reservoir allows to empty the reservoir entirely. During operation is has to be kept closed with the provided plug.

### 4.1.4. Pump

Regardless of the following instructions for operating the pump, the separate operating instructions of the pump manufacturer (KNF) must be observed.

The integrated pump system consists out of three components, each one of them is existential for a proper functioning of the whole system:

1. **Pre-filter**

To protect the pump against possible particles in the fluid, a prefilter (Material: PEEK) is used. A mesh size of 35 µm permits a reasonable flow rate under while maintaining its protective role.

2. **Pump**
3. **Pressure-sustaining valve**

A pressure-sustaining valve is used on the pumps pressure-side, to sustain a constant system pressure (650 mbar), to ensure a maximum accuracy during the test.

### 4.1.5. Connection of tube and prefilter to pump

Prior connecting tubes to pump, tube endings must be cap off flat with a suitable tube cutter. The tube must be clean and free of damages, to ensure a prober function of the attached pump. The prefilter should be replaced once a year.

### 4.1.6. Pump priming

Prior commissioning, the pump and inflow tube must be primed (Check manufacture manual for details). The system must be primed until no more bubbles are visible coming out of the pump. To facilitate the process pump can be tilt into all directions. By gently tapping the pump head and valve with pen (or similar), persistent air bubbles can be mobilized

### 4.1.7. Pump calibration

The pump has already been precisely adjusted in the factory. However, the device will operate even more precisely if it is calibrated. Calibration eliminates any variances caused by supply lines, back-pressure and viscosity.

To archive the highest possible accuracy, it is necessary to determine the flow rate by using a high-resolution balance. The following steps must be followed chronological, to calibrate the pump correctly and to make sure that the communication between pump and system control works probably:

#### Switch from automatic to manual mode

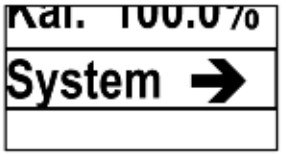
During the regular operation, the pump receives its control signal from the central control unit. In this mode, a large portion of the pump's functions are deactivated.

But the be able to calibrate the pump, this external control must be disabled.

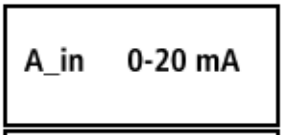
Use the control knob to move the selection display.


Pressing the knob confirms a selection

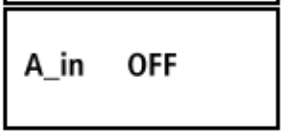

Go to the menu item **SYSTEM** and press the knob



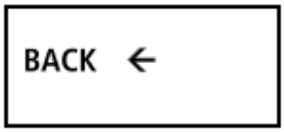

Go to the menu item **A_in**




Change from **0-20 mA** to **OFF** and press the knob (possible error message can be ignored)




Go to the menu item **BACK**




## Verification of flow rate

Go to displayed flow rate and press the knob. Enter the desired flow rate and press the knob. Choosen flow rate should correspond flow rate during leaching test

Make sure that the operation mode is "flow rate" by choosing **ml/min**

Activate the timeout function. The pump should run for ideally three minutes.


Press **START** button. The pump runs for the selected period of time with the defined flow rate.

This verification should be performed in case of doubt several times.

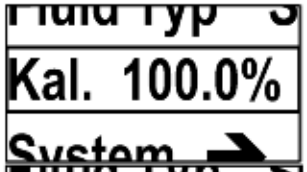
It has been shown that the first measured flow rate is often not correct. Best practice is to confirm a correct result by at least two consecutive measurements.

### Calibration

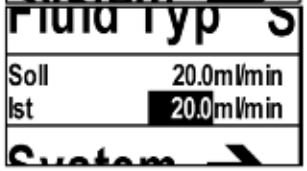
Depending on the chosen test, runtimes of several days and weeks can result. Even small deviations in the actual flow rate can risk the proper test implementation.

If deviations are noticed during the verification step, a pump calibration is necessary.

Go to the menu item **CAL. xxx %** and press the knob



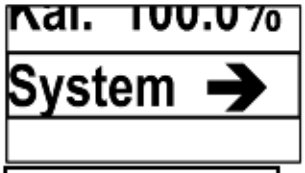

Enter the measured actual flow rate determined during the verification measurement and press the knob
NOTE: After pressing the know the main menu should appear. If not, do not enter the measured flow rate again. Otherwise the calibration would be incorrect. In case of doubt, a control measurement should be performed.



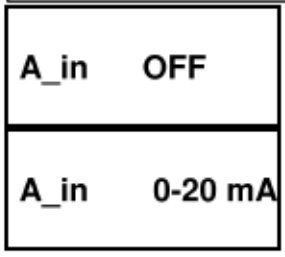

The new calibration should be verified by a control measurement.

### Reset from manual to automatic mode

Go to the menu item **SYSTEM** and press the knob




Go to the menu item **A_in**




Change from **OFF** to **0-20 mA** and press the knob (possible error message can be ignored)

Go to the menu item **BACK**

Press **START** button. Pump is now ready to start and can be controlled by the central control unit now


### Regular pump operation

During the automatic mode, the pump flow rate is controlled by the central control unit and cannot be manipulated by the user.

### Possible error sources

External sources can have a large impact on the flow rate. To archive the highest possible accuracy the following points should be noted:

- The installation location of the system should be as temperature constant as possible. Daily temperature fluctuations should be less than 2 ° C.
- The system should be operated with demineralize water. Ideally, the eluent should be degassed to avoid possible subsequent degassing in the system. When using other eluents, it is essential to consult the manufacturer in order to avoid possible problems due to deposits, contamination, etc.
- The eluent should only be tempered passively, which means that before use, the fluid should be at the installation site for at least 24 hours to acclimatize.
- The pump settings should not be changed (See page 44 for details)
- Tube length, diameter or material of suction tube should not be changed.

## 4.1.8. Saturation sensor

The sensitivity of the saturation sensor has to be verified once in a while to avoid malfunction.

### Sensor verification

As long as liquid is in the part of the tube that is placed right in front of the sensor, the LED control light should be yellow. Once the section of the tube is filled with air, it should light green.

Green light: Sensor is not active (e.g. air is in the tube)

Yellow light: Sensor is active (e.g. liquid is in the tube)

If this does not work, a readjustment of the sensor is needed.

### Sensor readjustment

The sensitivity of the sensor is adjustable by a slotted screw next to the control LED. Before the readjustment, make sure that the tube is clean and free of any residues.

*Figure 6: Saturation sensor with control LED*

Clockwise rotation increases the sensitivity of the sensor, counterclockwise rotation decreases it accordingly. When setting the sensor, a certain inertia of the device when switching from one state to the other (Active ↔ Inactive) should be taken into account (1-2 seconds).

Note: If the sensor is set too sensitive, there is the possibility that touching the sensor holder may even cause it to trip. On the other hand, if the sensor is set too insensitive, it can happen that the liquid flowing by is not detected. In both cases, the correct test procedure would be at risk.

## 4.2. AIO-Control7: Data input and system control

For data entry and system control, the PC control program AIO-Control7 and the control module are required as basic elements. The PC program is used for data entry, calculation of the experimental parameters and monitoring of the percolation process. The control module takes over the actual control of the process flow. An operating display on the control module additionally provides a choice between automatic mode for a PC-independent percolation process and manual mode with its subprograms **VALVES**, **PUMP** and **RINSE**.

### 4.2.1. System requirements and installation

The PC control software requires at least Windows 7 to function. It is computer-specific license-protected. Corresponding unlock codes are available upon on request.
To connect the PC with the control unit, the computer needs to be equipped with an individual network interface.

## Notes regarding first time installation:

Administrator rights are required to install the software (AIO-Control Setup.exe). The control unit has its own fixed IP address. Please discuss this with your IT administrator before installation!

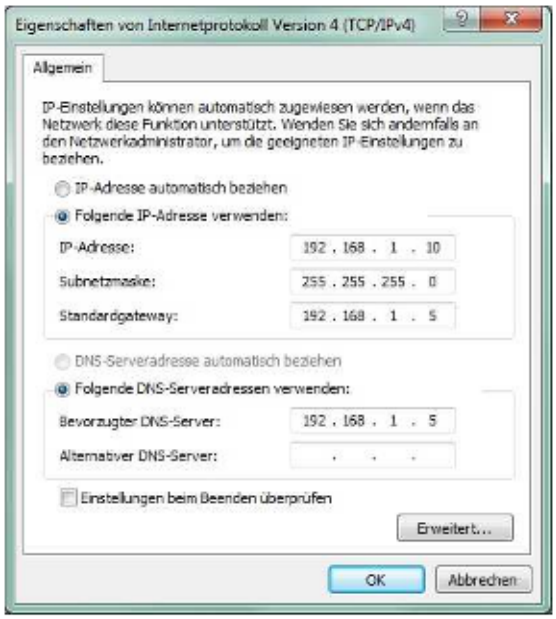



*Figure 7: Default IP address*

An installation routine automatically guides the user through the installation process. Make sure that the option **AIO-Control Full Installation** (Figure 8) is selected during the initial installation:

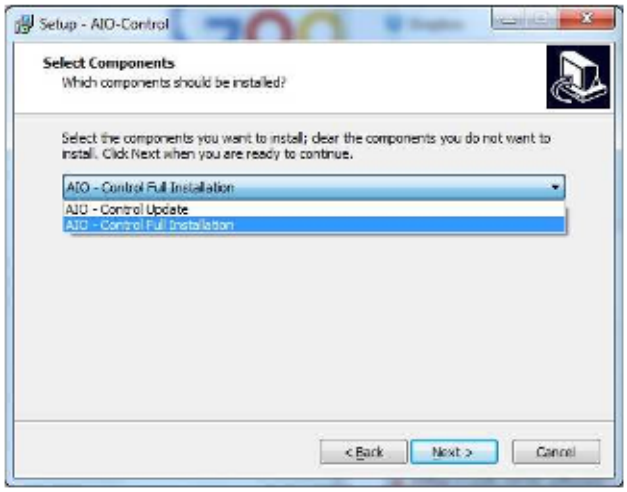



*Figure 8: Installation window*

After completing the installation, an icon automatically appears on the desktop to start the program:

After installing and starting the program for the first time, an activation window appears. Please create a screenshot from this window and send it by e-mail to us (soil@ecotech-bonn.de) by following these steps:

- Press the **PRINT** button on your keyboard after the unlock code entry window appears.
- Open your mail program and address a new message to soil@ecotech-bonn.de. Insert the screenshot into the text field with the command **INSERT** or **PASTE** (or key combination CTRL + V). This e-mail can be sent to us without any comments.
- An individual activation code will be sent back to you after a short time of processing (Usually within 24 hours). This activation code has to be entered once. To do so, open AIO-Control7 and enter it into the free white fields and press **ENTER**.

### 4.2.2. Program structure

The software-user interface is divided into the three areas: header, footer and main area:

#### Header

The header is permanently visible and subdivided into two sub-lines with three fields each (Figure 9).
In the first subline, these fields have the following functions:
Field 1: Current time and date
Field 2: Information about the currently selected menu
Field 3: Display of the submenu item (if required)


*Figure 9: Program header*

The display fields in the second subline have the following functions:

Field 1: "Heartbeat" of the program. Correct operation of the application is indicated by the dynamic running of the string *ASPA - Automated Column Percolation System*

Field 2: Displays malfunctions of individual components. All errors are archived in the "Alarm management" menu

Field 3: Communication status between the computer and the control unit.

## Footer

Like the header, the footer is also permanently visible and divided into several display fields. Some of them are dynamic, that means that they have change their content depending on the chosen active program section:


*Figure 10: Program footer*

These buttons have the following functions:

**Language** The software is designed for international use and has a multi-language pack. By default, the following languages are installed by default:

German

English

Further languages are available on request.

**Help** Opens this manual

**Print** Creates a screenshot and saves it in the program directory (.. \ PRINT)

**Alarm** Opens the module alarm management (s.u., Kap. 6.2.1.2.3.)

**System overview** Opens the module system overview

**Main menu** Goes back to main menu

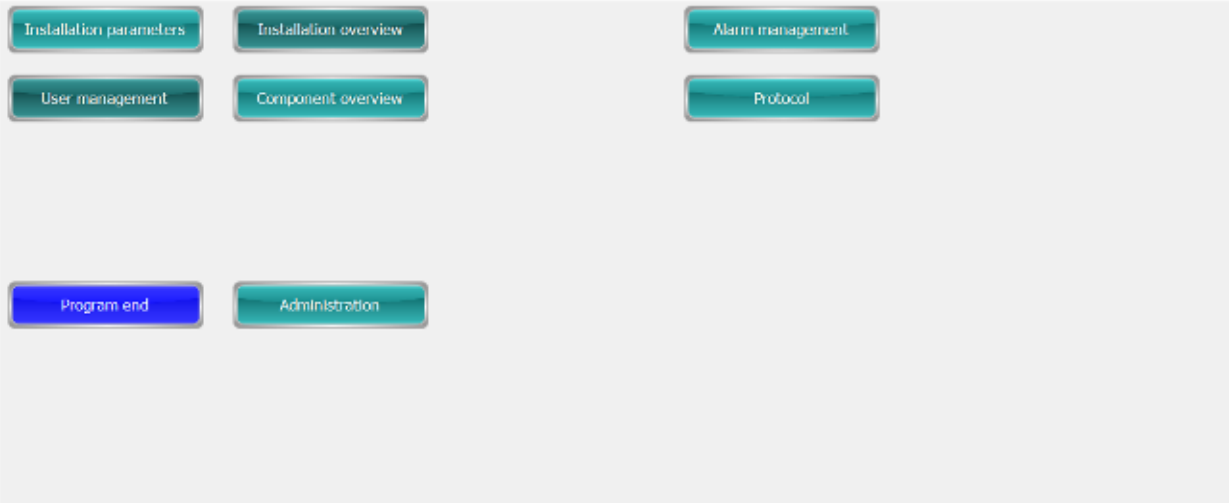



*Figure 11: Program main section*

**Main section**

The content of the central main section is dynamic. That means that its content changes depending on the chosen active program section.

### 4.2.3. Program sequence

Before starting the program, make sure that the control unit is already running.

During startup, the program automatically connects to the control unit. A progress bar shows this procedure. Once the communication between these two units has been initialized, the color of the communication field (top right of the screen) changes from yellow to green.

The main menu is the starting point to control the system. The following menu points are available:

| | |
|---|---|
| **System parameterization** | No access for user |
| **User management** | User must log in here before using the system. Without active log in, no tests can be started or canceled |
| **System overview** | Current status of all lines is visualized |
| **Component overview** | Current status of selected line (= individual leaching column with related sensor, pump and valves) all lines is visualized |
| **Administration** | No access for user |
| **Alarm management** | In this section, all system faults are managed and displayed as an alarm message in the header. Since the display field permits |

| | |
|---|---|
| | only one alarm message, the most recent alarm message is always displayed if several faults occur at the same time |
| **Protocol** | All actions carried out by the user are logged in the part |
| **End** | Ends the program after additional demand. Without prior log in, termination of the program is not possible |

### 4.2.4. User management

After opening this menu, all already created users are displayed. The user can log in or out via the **FUNCTIONS** button in the footer.

### 4.2.5. System overview


*Figure 12:* ***Installation overview***

This view can be accessed via the corresponding button in the footer or from the main menu.

The following parameters are displayed for each line:

1. Column number
2. Mode of operation (Manual or automatic)
3. Status
4. Incidents
5. Remaining overall run-time

6. Remaining run-time for active fraction
7. Current pressure in leaching column
8. Current nominal pump flow rate

Status colors are indicating the state of various individual components, green-marked elements are active:

9. Flushing valve
10. Fraction
11. Column saturation
12. Operation state of overall system
13. Pump

The calculated switching times (i.e., end time of fraction) of each individual fraction is also displayed. It must be pointed out that these times are purely informative at the start of the test.

The actual start of the test, defined by the time of complete saturation, is determined by the saturation sensor located at the hydraulic distributor. As soon as leachate in the tube is detected at this point, the column is considered as fully saturated, all dependent switching times are corrected accordingly once the moment comes.

By clicking on one of the displayed columns, the detailed view (= component overview) of the corresponding column opens.

### 4.2.6. Component overview

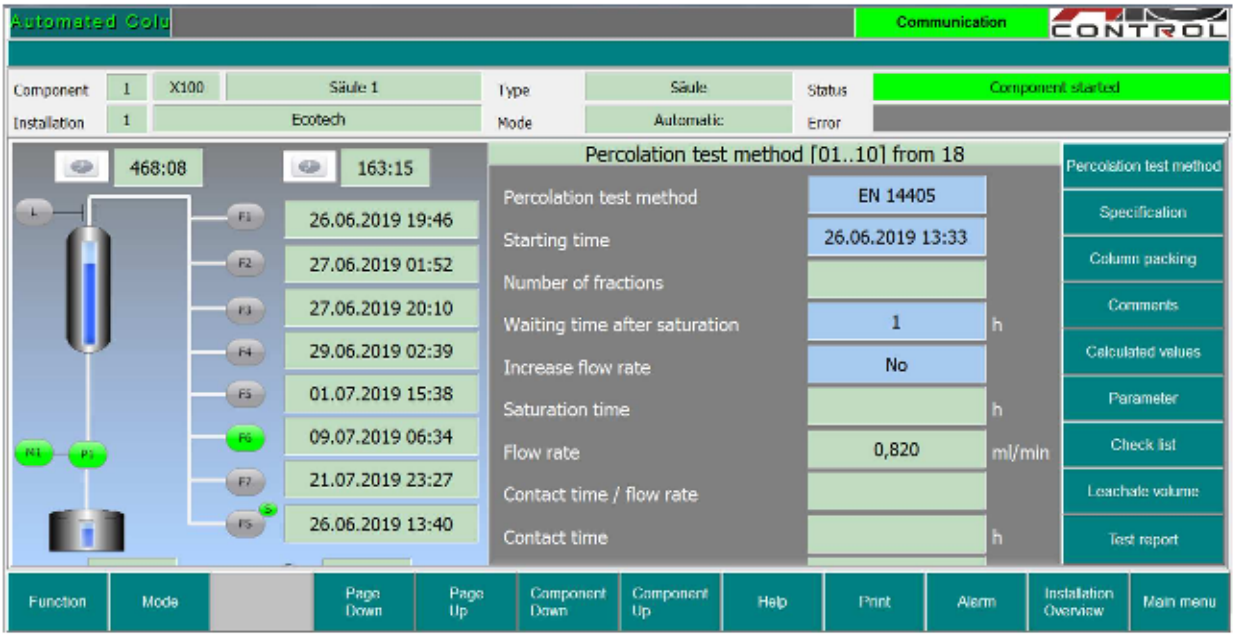



*Figure 13: Tab **Test methods***

This menu provides a detailed view of each column with all the parameters and functions necessary for operation. All actual values of the selected column are displayed on the left side of the. The middle part of the screen is filled with information depending on the function tabs on the right edge of the screen. It should be noted that field with a light blue background are editable, i.e. Information can be entered here. Non-editable fields are highlighted in light green.

If there are more parameters than can be displayed on one page, it will be indicated by the title, e.g. ... [0 ... 10] of 18 (See Fig. 28). The functions Page Down and Page Up in the footer, allow to switch between the pages.

The tabs are arranged to guide the user step-by-step from top to bottom through the process. By clicking on the individual tabs on the right edge of the screen, it is possible to switch between the individual screens as desired.

### 4.2.7. Test methods

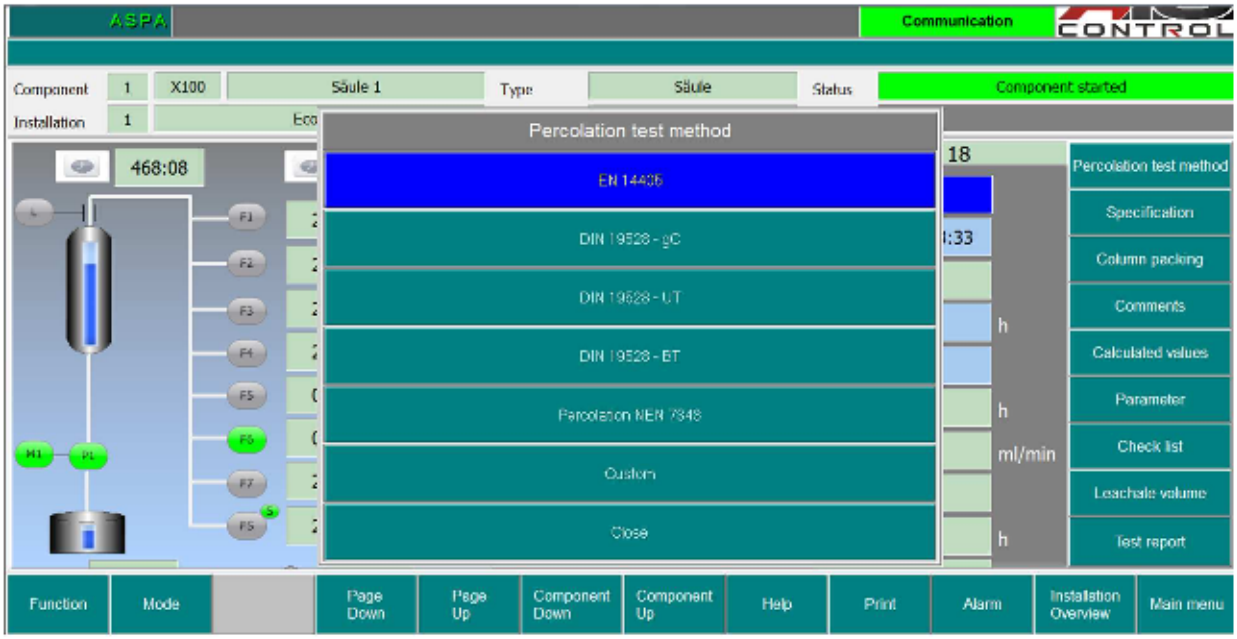



*Figure 14: Tab **Test methods**. Selection window for implemented test methods.*

At this point, the desired test method has to be chosen. The following tests are currently implemented, the names in brackets correspond to the nomenclature in the program:

- Basic characterization according to DIN 19528:2009 (DIN 19528 – gC)
- Compliance test according to DIN 19528:2009 (DIN 19528 – UT)
- Blank value test according to DIN 19528:2009 (DIN 19528 – BT)
- NEN 7373 (NEN 7373)
- EN 14405:2017 (EN 14405)
- Custom percolation test

The contact times and duration of each individual fraction resulting from the selected test method are automatically determined.

In the percolation method tab, the start time for the test must be set by the user. After clicking on the **START TIME** button, a separate window opens. The user has the option to select the current time directly as the start time. To do so, the button **RUN IMMEDIATELY** has to be pressed. Alternatively, it is also possible to program a start time in the future. To do so, a time and data on the open calendar page has to be selected and confirmed with **EXECUTE**.

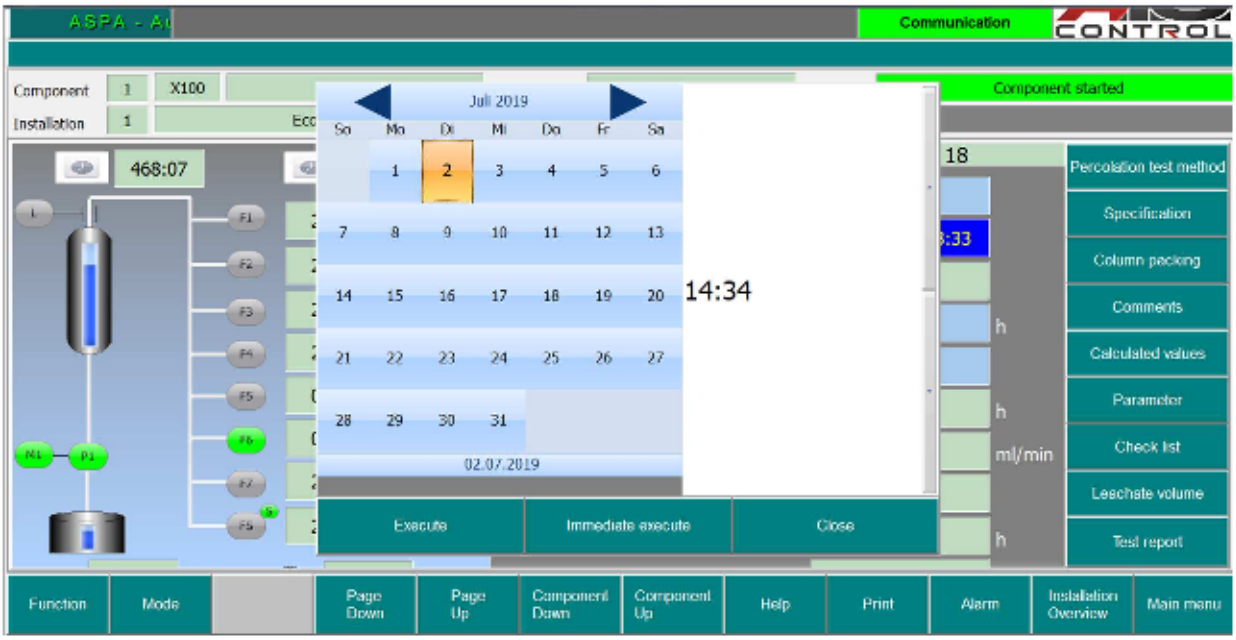



*Figure 15: Tab test methods. Selection field Starting time*

In both cases, however, the system does not start immediately, but must be started separately by the user in a last step.

When choosing the test method **CUSTOM PERCOLATION TEST** the user must define the followings parameters before being able to start the test:

| **Parameter** | **Description** |
|---|---|
| Number of fractions | Number of fractions to be collected. Maximum number depend on hydraulic distributor (4 or 7, respectively) |
| Waiting period after saturation | Waiting period after the column is completely saturated. The pump is not running and all valves are closed. After the defined period, the test starts automatically. |
| Duration of saturation/ Flow rate | The column saturation rate can be entered indirectly by entering a desired saturation time or directly by entering a defined flow rate. Depending on the selected parameter, one of the two following fields becomes editable. |
| Duration of saturation | see above |
| Flow rate | see above |
| Contact time/ Flow rate | The flow rate during the test can be entered indirectly by entering a desired contact time between sample material and eluent or directly by entering flow rate. Depending on the selected parameter, one of the following fields becomes editable. |

| | |
|---|---|
| Contact time | See above |
| Flow rate | See above |
| PV/LS | The fractionation of the leachate can be calculated on the base of the pore volume (PV) or on the liquid/solid ratio (LS) |
| Coefficient 1-7 | Target values (corresponding to PV or LS; depening on the above selected parameter) for each individual fraction. Entered values must be ascending. |

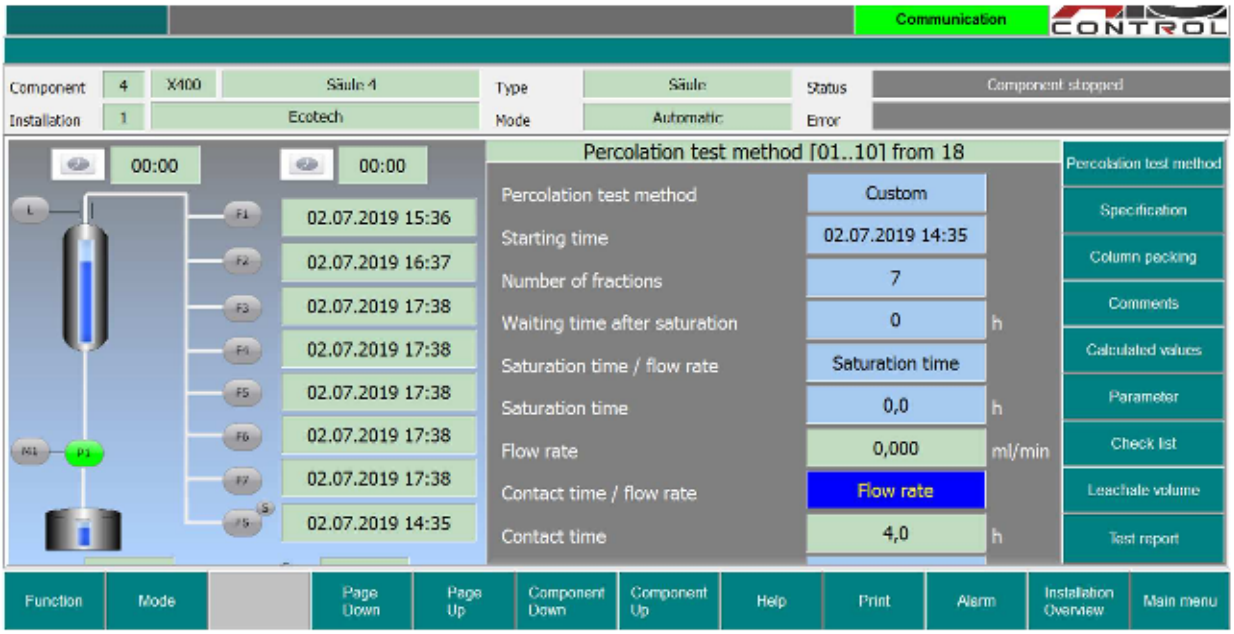



*Figure 16: Percolation test method II*

### 4.2.8. Specification

In this tab all specifications regarding the sample type, its origin and material must be entered:

- Ambient temperature
- Sample number
- Sample origin
- Sample description
- Column material
- Filling height of sample
- Column inner diameter
- Tube material
- Filter material
- Preconditioning of filter material
- Grain size of filter material
- Method in Installation

### 4.2.9. Column packing

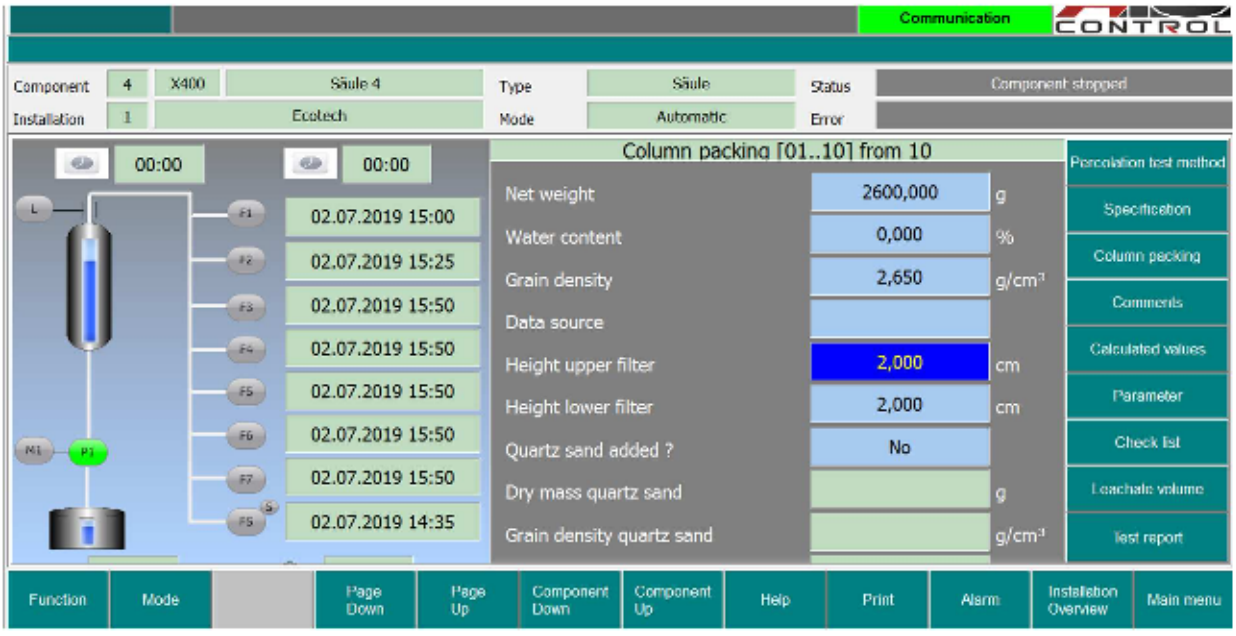



*Figure 17: Tab column packing*

In this tap all basic parameters to determine the pore volume of the sample have to be entered, the program derives the flow-through time and leachate volume from these data.

As an addition to the parameter **GRAIN DENSITY**, the following line **DATA ORIGIN** must be used to specify whether the specified value was measured directly or was taken from a table.

In the input field "Filter material" the height of the upper and lower filter bed has to be entered (if used). To calculate the saturation time for the whole leaching column later on, a porosity of 0,4 for the filter bed is assumed since the grain size of the material for the filter should be between 0,6-1,2 mm according to DIN 19528.

The field "Quartz sand amendment" is not activated by default. It is only needed when cohesive samples with low hydraulic conductivity should be tested and additional quartz sand has to be added (according DIN 19528). The NEN 7373 percolation test does not mention this possibility. When activating "Quartz sand amendment" the program assumes automatically an addition of 80 % quartz sand to the total quantity.

### 4.2.10. Comments

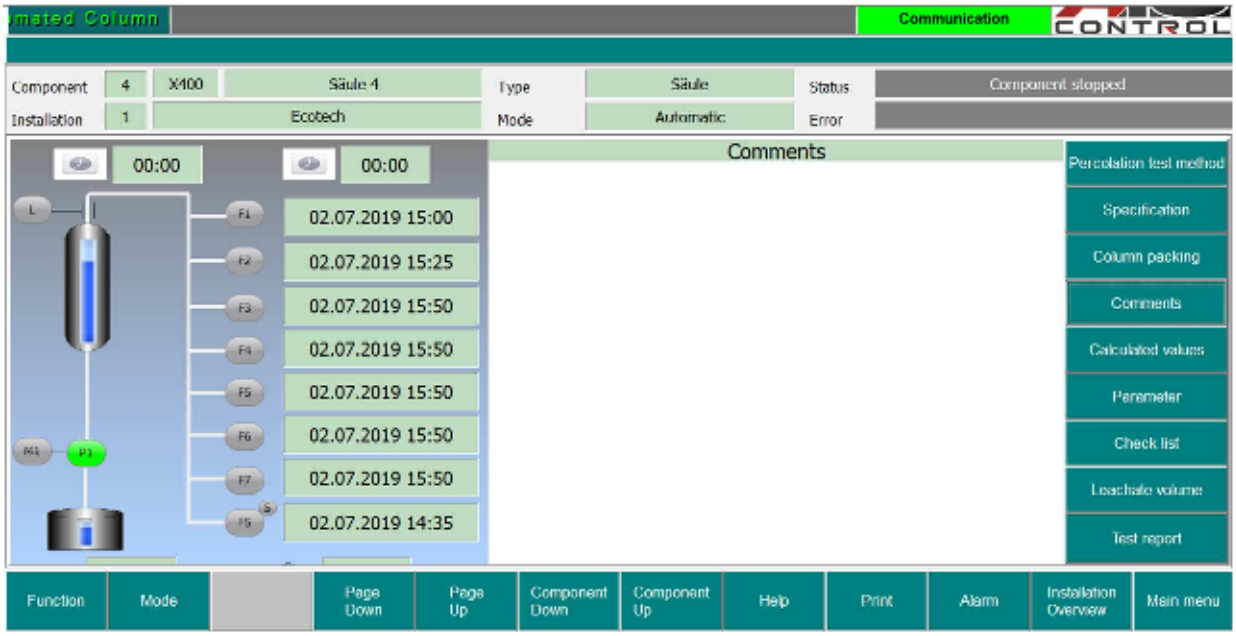



*Figure 18: Tab comments*

In this field, comments can be entered, even after completion of a test run, and will be displayed on the latter lab report.

## 4.2.11. Calculated values

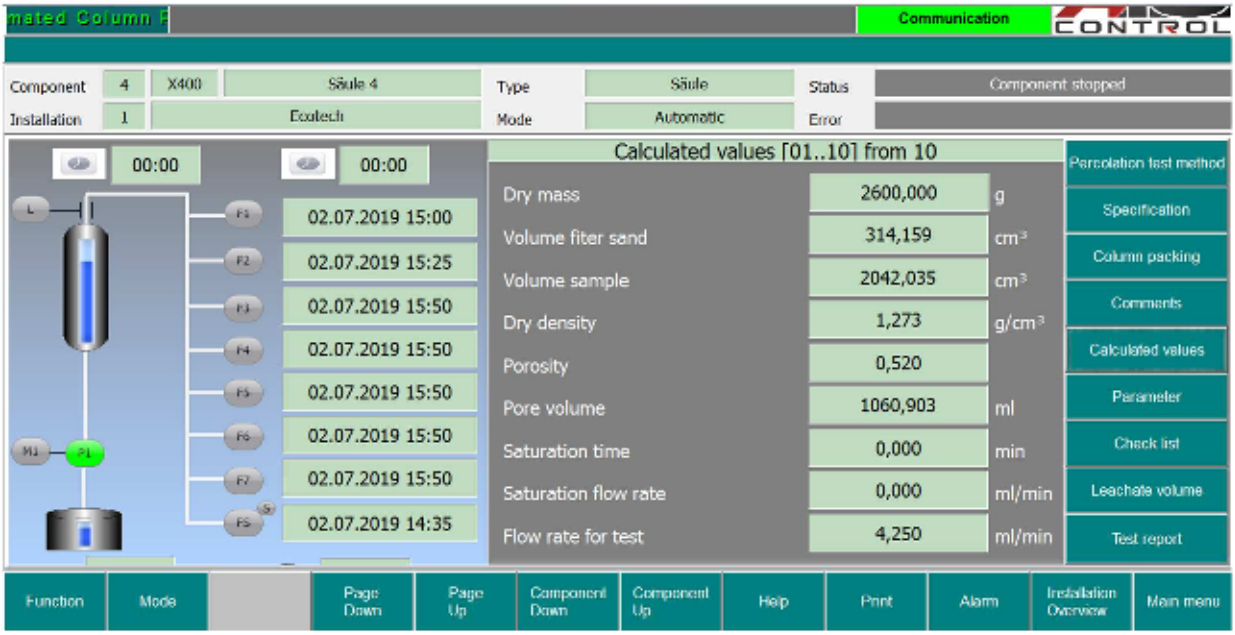



*Figure 19: Tab Calculated values*

The tab **CALCULATED VALUES** contains the calculated values based on the previous inputs. At this point, no entries or changes can be made. Please check at this point, if all values are displayed plausibly. If this is not the case, please check your entries again and correct them accordingly.

Before each test, the pump should be calibrated to the value **FLOW RATE AT TEST**, details of the individual steps can be found in chapter 4.1.7 from page 13 onwards.

## 4.2.12. Parameter

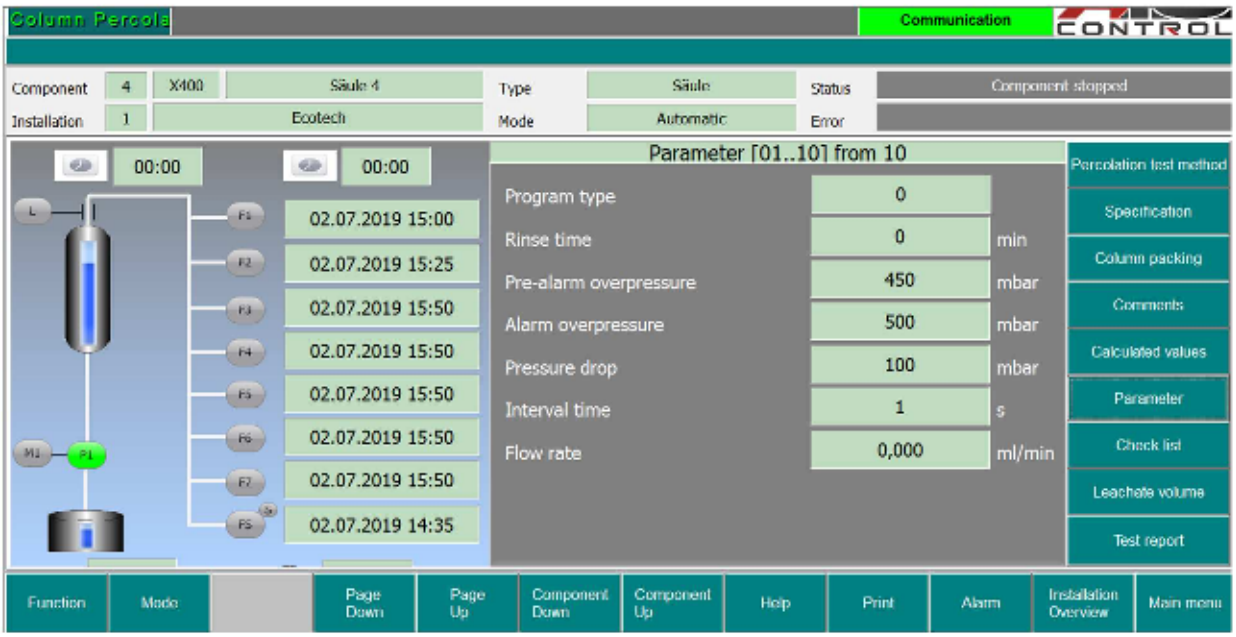



*Figure 20: Tab Parameter*

Depending on whether the system is in automatic or manual mode, different entries can be made here:

System is in automatic mode:

Limit values for pressure monitoring of the systems during automatic operation

System is in manual mode:

Choice of the desired program:

**VALVES**

**PUMP**

Flow rate (0-20 ml / min)

**RINSE**

Purge time (minutes per valve)

### 4.2.13. Check list

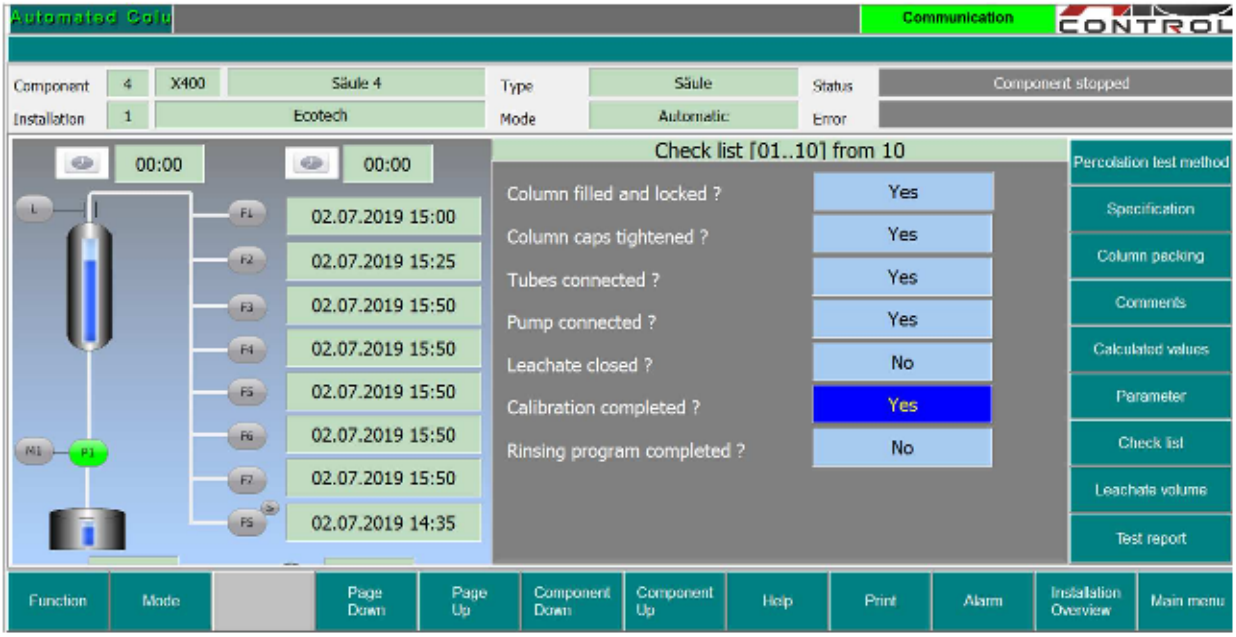



*Figure 21: Tab Check list*

This is the last tap to be used before starting the test. Several safety questions have to be answered. They concern the mechanical securing of the columns and the distribution system of the sampling bottles. This checklist refers only as references.

If necessary, the starting time can be corrected for the last time, before the test is started via the function key in the footer of the program. If necessary, the test can also be canceled with this function.

### 4.2.14. Leachate volume

After the end of the test, the measured eluate volumes have to be entered in this tab. The entered values appear later in the test report as reference values.

## 4.2.15. Test report

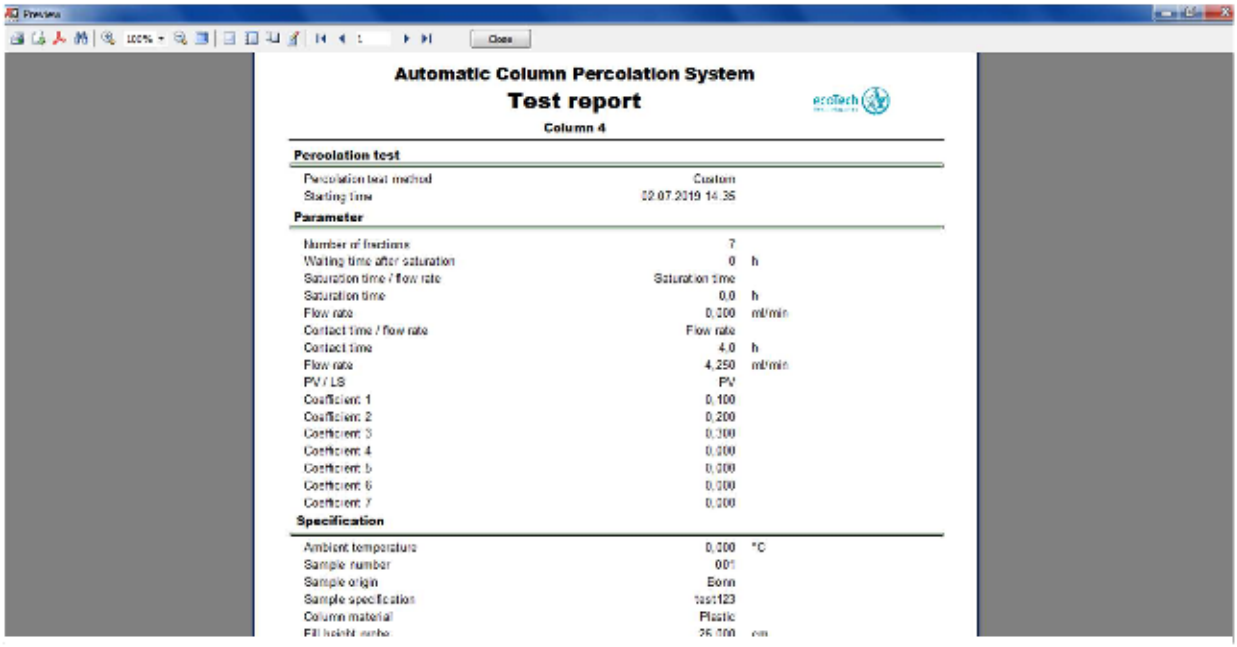



*Figure 22: Preview Test report*

When selecting this tab, a window appears, the function **DISPLAY** calls up the test report, the function **DELETE ALL PARAMETERS (COMPLETE)** deletes all entered parameters and resets the system!

The test report contains all the information required by DIN for the proper performance of a column percolation. These are, in particular, the type of percolation, the information on the sampled substrate and the incorporation process, the volumes of the eluate fractions and the flow times. The test report also contains comments and explanations on the course of events or deviations within the percolation passage.

For documentation, this report can be printed directly, saved as a PDF file and exported as spreadsheet.

After generating the test report, the system must be reset via the function **DELETE ALL PARAMETERS (COMPLETE)** in order to be ready for the next test.

## 5. Control unit

Besides the PC program, the control unit is another important interface for the user. The PC program sends the calculated process parameter to the controller; after that moment, the controller assumes all control function and executes the test automatically. From this point on, the computer has only a monitoring function. The control unit is switched on or off by pressing the main switch on the control cabinet

*Figure 23: Control unit with and display and main switch*

After a successful boot process, the main menu opens on the operating display, where all leaching columns are displayed, with the following information:

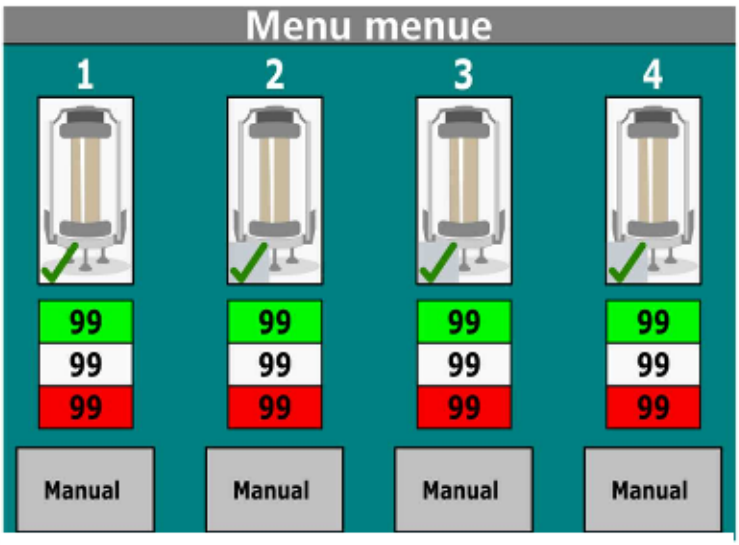



*Figure 24: Menu menue*

Every leaching column can be operated in two different modes: Manual or automatic. The selection of the operating mode is made via the bottom button on the display.

In the manual mode, several subprograms can be started, which are necessary for the preparation of a percolation test. These are applications require control by the user. The automatic mode is used to automatically perform column percolation test.

These two modes are explained in detail below:

By tapping the column icon, an integrated button is activated. If the icon is selected while the column is in automatic mode, the sub-program status (Figure 26 on page 37) is activated. If the manual mode is active, the subroutine manual programs (see below) opens.

### 5.1. Manual programs

In this sub-program, the following options exist:

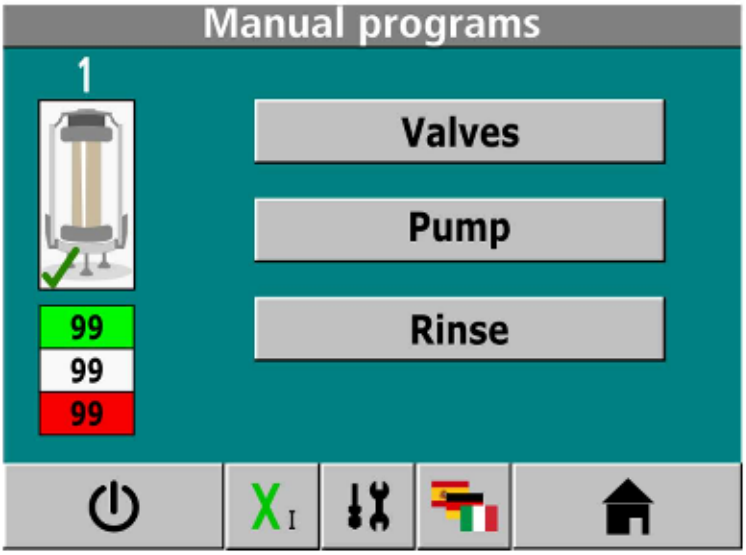



*Figure 25: Subroutine **Manual programs***

**Note:**

Theses subprograms can only be operated individually and not parallelly. If one program is active, all others are blocked. They do not stop automatically but must be terminated by the user.

The screen footer displays the following buttons:

- Start/Stop of chosen program
- Status of the chosen column
- Setup menu of the chosen column. Opens the second page of the menu.
- Language
- Home

### 5.1.1. Valves

By choosing the program **VALVES** and activating it via the **START/STOP** menu, all valves on the hydraulic distributor are opened at the same time.

This function is used after the text finishes, to obtain complete samples by emptying all tubes. Before opening the valves, it is necessary to separate the column outlet from the hydraulic distributor in order to allow a pressure compensation.

### 5.1.2. Pump

By choosing and activating the program **PUMP**, connected pump starts to work. Prior activation, the flow rate has to be defined in the menu item **SETUP**.

### 5.1.3. Rinse

This program is used to clean the tubing connected to the hydraulic distributor. When activated, the connected pump runs at the speed defined in the setup menu, and all valves are opened one after the other. The duration of the rinse cycle can also be defined by the user via the setup menu.

It is advisable to perform a blind value test after the rinsing process. If it turns out that the cleaning did not have sufficient effect, the process must be repeated, or the tube system should be replaced.

## 5.2. Automatic mode

In order to carry out a column percolation test, the system must be put into its automatic mode. This can be done directly through the control unit or through the PC control software AIO7 (footer). After entering all necessary data, the percolation process can be started via the PC or via the service display on the control module.

The start button can be found in the PC program AIO-7 in the program footer of the button function. This option always to start or to stop ongoing tests.

To start a test from the control unit, the Start/Stop button has to be used. The same button also stops an ongoing tests.

After the start, all important process information of running column is displayed in the column status menu (Figure 44). The same information is also presented in the PC software.

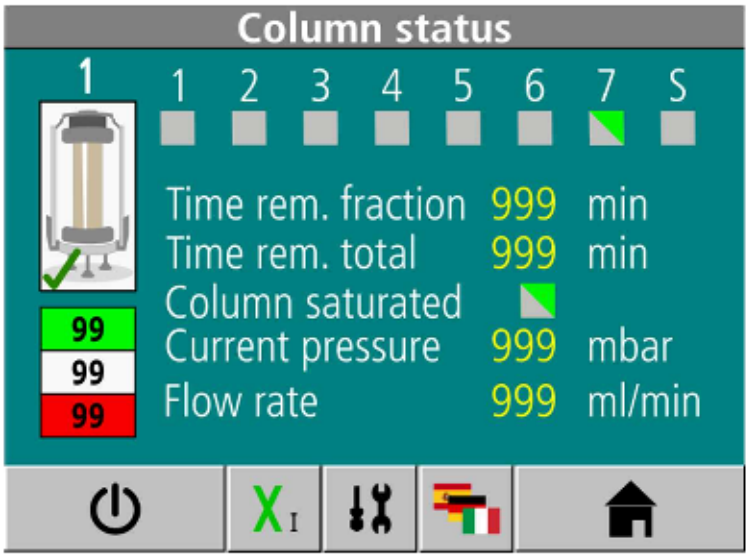



*Figure 26:Menu* ***Column status***

The status of each fraction is displayed on top of the graph, active components are underlaid in green. Below, the remaining time for the active faction, as well as for the overall test in minutes is displayed. As soon the leaching column is fully saturated and leachate starts to leak from the column outlet, the corresponding display turns green. The current pressure in the system, as well as pump flow rate, are also shown in this screen.

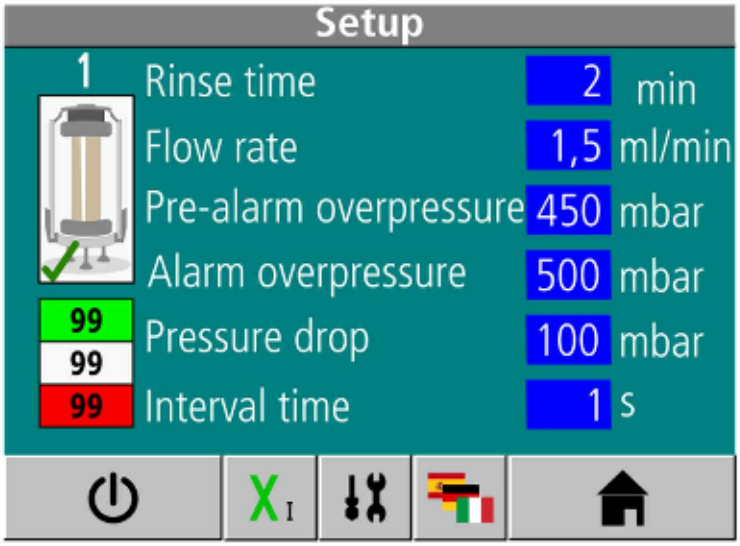
After the test ends, the pump stops automatically and all valves close. In order to empty all tubes completely to a complete sample of each individual fraction, all valves can be opened for a short time using the program **VALVES.**

The determined volumes of the individual fractions can then be entered into the corresponding input mask of the PC Software AIO-Control7 and the test can be terminated with the preparation of a test report.

The determined volumes of each individual fraction can then be entered into the corresponding input mask of the PC Software AIO-Control7 and a lab report can be created.

### Alarm messages during automatic mode

During automatic operation, the system pressure between the pump and the column is constantly monitored. It is necessary to prevent a pressure build-up due to swelling material, too low hydraulic conductivity or clogging of pores.




The following thresholds are set-up by default to ensure a proper operation of the system:

Pre-alarm: 450 mbar

Main alarm: 500 mbar

The pre-alarm is used to alert the user of the attachment to alert him to an existing problem. The operation of the system is still possible with this system pressure.

Once the main alarm is exceeded, the system will run for another 10 minutes, before it stops automatically. During this time, the display shows **FAILURE**. If it is possible, to solve this problem during this period of time, it is possible to quit this failure. In that case, the failure is repealed, and the system will continue working.

**Note:**

Even in the event of sudden pressure loss, the system goes into an alarm mode, as this sudden change is interpreted as a possible leakage in the system.

By default, the following limits are preset:

Pressure drop: 100 mbar

Interval time: 1 s

Following this set-up, the system goes into alarm mode if the pressure drops by 100 mbar within one second. The pump will stop and all valves will close.

Upcoming alarms are shown in the PC software in the header and as well as in the program **COMPONENT OVERVIEW**. The alarm is also displayed on the operating display.

After the malfunction has been removed, the error message needs to be acknowledged. This is done via the menu **ALARM** in the footer. In this menu, the alarm can be used to acknowledge the status of the system back to normal. For safety reasons, the alarm must also be acknowledged directly to the control unit.

## 6. Troubleshooting

| Problem | Solution |
|---|---|
| The operating display shows **FAULT** | The pressure inside one or more leaching columns is/was too high. Please eliminate the cause (eg hosed or pinched hoses). The system switches off after 10 minutes in alarm mode!<br>If the substrate is not suitable due to a low hydraulic, it must be mixed with 80% quartz sand (see DIN 19528). |
| Pump does not start | Please check, if the pump is set to automatic mode. |
| Leaching column is leaking | After leaking, remove the upper closure of the column and dry all parts (sealing, column, clamping ring, clamping ring) and reconnect everything. The clamp should always be used to increase the pressure tightness of the columns. |
| AIO-Control7 does not allow data entries | Check if you are logged in as user in AIO-Control7. |
| Actual fractional volumes deviate from calculated volumes | Verify the pump flow rate of the pump, if necessary, the pump must be calibrated.<br>Air bubbles in the pump head also lead to shortage. During operation of the pump, tilt the unit to improve ventilation. |

## 7. Literature

DIN 19528 (2009): Elution von Feststoffen - Perkolationsverfahren zur gemeinsamen Untersuchung des Elutionsverhaltens von anorganischen und organischen Stoffen

FprCEN ISO/TS 21268-3 (2009): (D) Bodenbeschaffenheit - Eluierungsverfahren für die anschließende chemische und ökotoxikologische Untersuchung von Boden und von Bodenmaterialien – Teil 3: Perkolationstest im Aufwärtsstrom (ISO/TS 21286.3:2007)

Kalbe, U.; Berger, W.; Simon, F.G. (2008): Abschlussbericht zum UFOPLAN FuE-Vorhaben Durchführung von Ringversuchen zur Validierung der Normen E DIN 19528 und E DIN 19529 (Ersatzbaustoffverordnung und No-vellierung BBodSchV), Bundesanstalt für Materialforschung und –prüfung (BAM), Berlin

Kalbe, U.; Berger, W.; Simon, F.G. (2009): Praktische Aspekte der Anwendung von Eluierungsverfahren nach DIN 19528 und DIN 19529 In: Altlasten Spektrum. Ausgabe 1

Landesumweltamt Nordrhein-Westfalen (2000): Empfehlungen für die Durchführung und Auswertung von Säu-lenversuchen gemäß Bundes-Bodenschutz- und Altlastenverordnung (BBodSchV), Merkblätter, Nr. 20, Essen

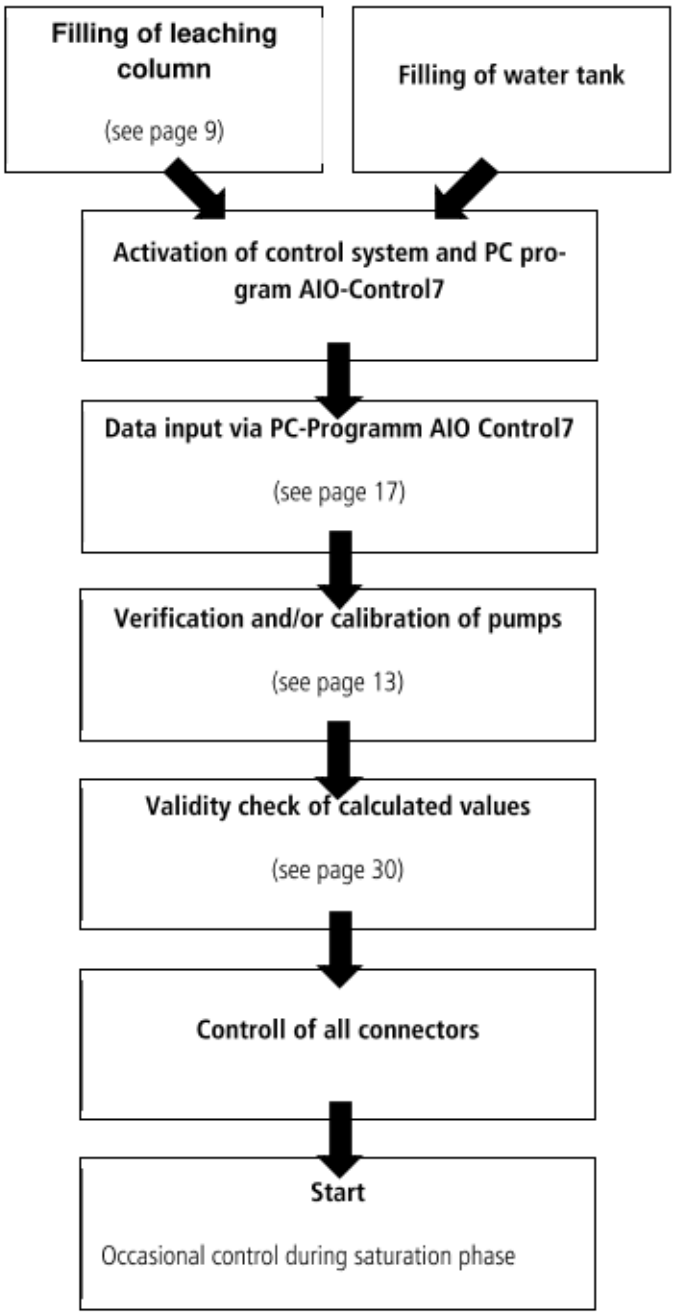
Lewis, J.; Sjöstrom, J. (2010): Optimizing the experimental design of soil columns in saturated and unsaturated transport experiments. Review article. In: Journal of Contaminant Hydrology 115. 1-13. Elsevier

Strasser, H. (1993): Grundsätze der Arbeitsplatzgestaltung; In: Hettinger, Th. und Wobbe, G. (Hrsg.): Kompen-dium der Arbeitswissenschaft. Kiehl-Verlag, Ludwigshafen/Rhein, 179-191

# 8. Annex

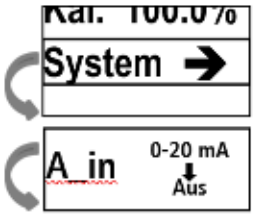
## 8.1. Quick guides

### 8.1.1. Process flow

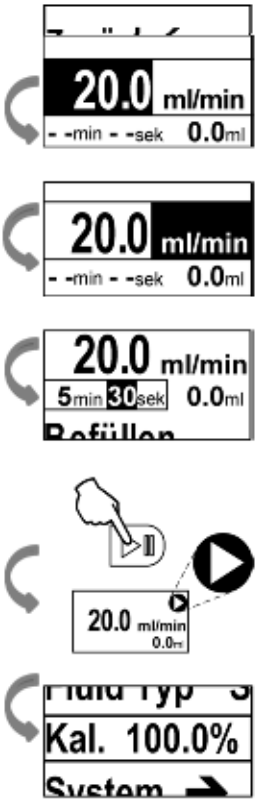


### 8.1.2. Verification and calibration of pump flow rate

1. **Change from automatic to manual mode**




2. **Control of flow rate and (if necessary) calibration**



3. **Return to automatic mode**


### 8.1.3. Pump default setting

| | |
|---|---|
| Fluid type | D |
| A_in | 0-20 mA |
| Autostart | Ein |

### 8.1.4. Displayed status messages

**Program active**

**Program inactive**

Automatic mode

1. Valves
2. Pump
3. Rinse

**Existing start conditions**

**No existing start conditions**

No start conditions

1. No program selected
2. Valve number = 0
3. Maximum rpm = 0
4. Rinsing duration = 0
6. Time interval pressure loss = 0
7. Pressure pre-alarm = 0
8. Pressure drop alarm = 0
9. Pressure alarm = 0
11. Flow rate = 0
26. Communication interruption

Start conditions are checked in conditions of selected program

**As long as a start condition exist, the chosen program cannot be executed.**

**No pending alarm**

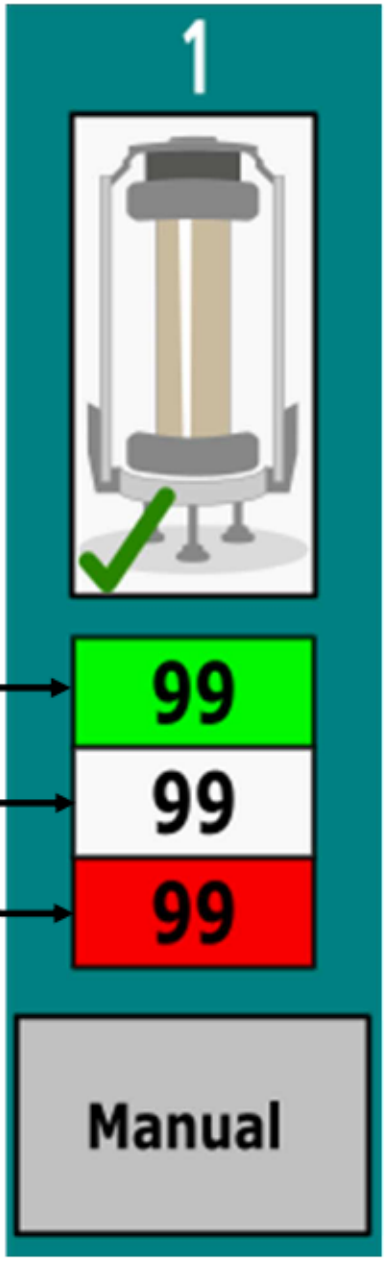
**Pre-alarm pending**

**Alarm pending**

No alarm

4. Pressure pre-alarm
5. Pressure alarm
6. Pressure drop alarm
14. Pressure alarm
16. Pressure drop alarm

**Pre/Alarm conditions are only monitored in the automatic mode**



## 9. Imprint

This system is a joint development of ecoTech Umwelt-Meßsysteme GmbH (Bonn) and the Federal Institute for Materials Research and Testing (Berlin)

The cooperation project was funded by the Federal Ministry of Economics and Technology within the framework of the " ZIM - Zentrales Innovationsprogramm Mittelstand / ZIM - Central Innovation Program for SMEs" from 15.02. 2010 to 15.08.2011.

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