LCTech

DEXTech Heat

Semi-Automated Sample Preparation for PCB and Dioxin
User Manual

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User Manual

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Dear customer,

We are delighted that you have chosen an LCTech product, and would like to thank you for the confidence that you have placed in our company.

We have developed this high-performance device with the utmost care and manufactured it using high-quality materials and componentry thus making it a reliable asset in support of you and in mastering your daily workload.

Please take time to read this manual completely and observe all the instructions given for both your own safety and to ensure the best possible handling and functionality of the device.

Our LCTech team will be happy to assist you. Please do not hesitate to contact us if you have any questions or require assistance.

We hope that you enjoy using your new DEXTech Heat.

Best wishes,

Your LCTech team

Content

Important Notes

I) Device Instruction

Please read these notes and the user manual before commissioning and using the device. Please follow all instructions given carefully.

Make sure that the system is installed and operated on a table with a minimum bearing load of (50 kg)!

Any improper operation, application, maintenance or repair may have fatal consequences. These apply to both the user and the system!

The system must NOT be used for:

- Unstable substances
- Substances that might explode upon mechanical impact or temperature elevation without additional airing
- Self-igniting substances
- Substances which are ignitable without additional air supply
- Explosives
- Acids

This device is not approved for installation and operation in hazardous areas or outdoors.

The system complies with the fundamental requirements regarding design and construction in accordance with the appropriate EC guidelines. These standards determine environmental conditions under which the system can be safely operated.

The system can be operated at environmental temperatures of +10 °C to +25 °C and a relative humidity of 10 % up to a maximum of 90 % non-condensing.

The system may be operated at altitudes from sea level up to 2000 m.

The electrical protection class is IP20.

Please ensure adequate ventilation, in particular in the area of the external power supply: Sufficient space must be left above and underneath the power supply for convection of cooling air. Do not cover the cooling slots!

Improper usage, disregard of safety instructions clearly outlined within this manual, misuse of the system, as well as inappropriate maintenance/repair by non-authorized service personnel or the use of other than genuine spare parts and accessories voids any claims against the manufacturer.

The individual components may be connected in the intended way only.

Attention: If the system in not used for some time (for example 2 days, the weekend or for transport) the column tower should not be closed completely, to remove the pressure from the dummy columns and to prevent misalignment of the sensor. To do so close the column tower completely using the “Close“-button. Afterwards open the column tower so far that the dummy columns can be moved slightly with your hand.

Never reach directly into moving machine parts or into gaps, which are intended for mechanical movements.

II) Electrical Instruction

ATTENTION: As with all mains powered devices, please observe the following safety precautions:

- Protect the system and the external power supply from any dripping or spilling liquids. There is danger of short-circuit or fire (protection class of the power supply is IP20).
- Do not make any inappropriate alterations to the system.
- Switch off the system in emergency cases (de-energized). 
- Operate the system under the given environmental conditions only.
- Operate the system with the rated voltage only: 100-240 Volt AC, 50-60 Hz.
- Replace broken fuses with the same type only.
- Operate the system with a functional ground pin.
- In order to increase your personal safety, an earth leakage circuit breaker with a cut-off current of 30 mA or less should be installed.
- Do not operate the system with broken power cables; the repair of these is mandatory.
- There are heated elements. Please note safety sticker.

III) Solvents: Handling of Solvents and Waste Disposal


ATTENTION: When handling chemicals, always wear protective gloves and goggles.

Collect the solvents in a suitable container only. The container should be sufficiently vented and have the intended, tight fitting lid.

The waste tube must be tightly fixed to the upper lid of the waste container; the outlet of the tube should sit just beneath the lid in order to guarantee proper function.

Avoid kinks, loops or coiling of the waste tube; or else the solvent may not drain properly. The maximum volume of the waste container must be sufficient to collect all solvent, which may be fed into the system as an eluent or cleaning agent.

Alternatively, using an LCTech waste level sensor is recommended.

If you are using the LCTech waste level sensor, please make sure that the waste tube does not get in contact with the floater. Avoid the waste tube of being immersed too deeply.

The waste container needs to be completely emptied before the system is refilled with solvents. When refilling solvents, please ensure that the quality used is suitable for the intended application. The solvent must be free of any particulate matter. Make sure the solvent tubings of the reservoir are free of bubbles.

If necessary, the tube can be vented via the vent outlet, before the pump is operated again.

Make sure the open end of the solvent tubes is at the bottom of the solvent supply bottle and not directed upwards. Using sinter filters is recommended.

Make sure any solvent supply bottles and waste containers are vented properly in order to prevent any vacuum formation and to ensure correct solvent delivery.

Where appropriate connect any filter or waste air tubing.

Disposal of wastes

Please observe local regulations for collection and disposal of laboratory waste as well as the relevant safety data sheets for the first column and the solvents used.

Please take note of the detailed explanations given in this user manual in the individual chapters.

This introductory chapter is intended to point out some particular circumstances only.
The whole procedure recommended by LCTech is explained in the following.

1. Intended Purpose

Major PCDD/F and PCB contamination cases, which have occurred in the food and feed chain over the last few years, illustrate the need for fast and high throughput methods to help identify and confirm non-compliant samples, which then can be traced back to the source of contamination.

For this purpose, a highly efficient clean-up procedure is required to purify raw extracts prior to the final analytical separation and quantification.

The DEXTech Heat system was developed based on the DEXTech Pure system. The system includes heating parts (sample vial holder, tubings, and the sample loop) for processing of difficult samples and fats that harden at room temperature. These include for example samples like palm oil, palm fatty acid distillates (PFAD), palm stearin, stearic acid or palmitic acid as well as different animal fats.

DEXTech Heat can also be used for the sample clean-up of Dioxins and PCBs in a variety of matrices such as:

- Food and feed samples, e.g. meat, fish, fish oil, eggs and various vegetable oils
- Environmental samples including sludge, soil and sediment samples
- Biological samples, e.g. blood

The system is equipped with heating parts for clean-up up of samples with a melting point higher than 25 °C. It keeps melted samples liquid from sample vial to loading onto the first column without any manual handling, as the system heats it at all necessary positions.




The default methods in the automated DEXTech Heat system are based on a 3 column set-up including sulfuric acid coated silica gel, alumina column and carbon column. The fractionation process of the LCTech automated clean-up system allows for rapid and separate analysis of PCDD/F, non-ortho, mono-ortho and ndl-PCB.

Gas chromatography with high resolution mass spectrometry (GC–HRMS) or gas chromatography with tandem mass spectrometry (GC-MS/MS) can be used for the analytical determination of the two fractions. A comparison study was performed on quality-control samples to evaluate the robustness of the automated sample clean-up system and also the quantification by GC-HRMS and GC-MS/MS. All results demonstrate the suitability of the automated LCTech sample preparation system and the GC-MS/MS system for fast and reliable routine analyses of PCDD/F and PCB congeners in foodstuffs and feedstuffs meeting the requirements of European Union legislation.

2. Environmental Conditions/Technical Data

2.1. Physical Data

Environmental temperature admissible during	
Storage:	0 °C + 50 °C
Operation:	+10 °C + 25 °C
Dimensions system W x H x D:	440 x 670 x 500 mm
Dimensions control unit W x H x D:	310 x 75 x 160 mm
Weight:	46 kg

2.2. Electrical Data

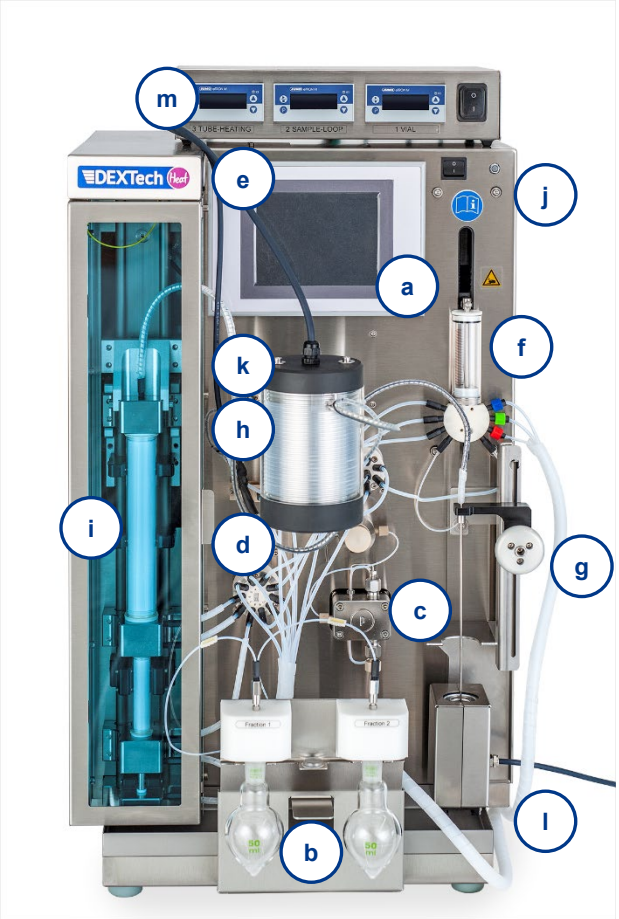
Protection class:	IP 20
Supply voltage:	100-240 Volt AC, 50-60 Hz
Maximum rating:	350 W
Internal fuse:	2 x F4A 5 x 20 mm 1 x F1A 5 x 20 mm
Internal voltage:	24 / 36 Volt DC

2.3. Process Engineering

Maximum pressure:	15 bar (217.7 psi) 5 bar for column 1
Pressure sensor:	0.1 – 15 bar (1.5 - 230 psi)
Minimum flow rate	0.1 mL/min
Maximum flow rate:	40 mL/min
Max. flow rate in process:	7 mL/min
Wetted parts:	Stainless steel PTFE Ceramic Glass PEEK
Gas supply:	Nitrogen (2.0-3.0 bar ~ 29-43 psi) supplied by lab
Max. syringe pump volume	25 mL

3. Explanation of the System Components

3.1. Front Side of the Device



(a) Touch screen

The touch screen is used for the control of the system. The surface is protected by a foil.

(b) Fraction rack (optional)

It is possible to fit two round bottom flasks (NS 14/23) for collecting fractions. This rack is mounted on the DEXTech Heat.

(c) Piston pump

The preparative pump is a double piston version in order to reduce pulsation. The pressure sensor is specifically adapted to the working range in order to provide an accurate reading.

(d) System valves

The valves control the entire liquid handling of the DEXTech Heat system.

(e) USB socket

The USB socket is used to transfer the sample data onto a USB stick. Afterwards, a pdf-report for each sample can be generated using special software on the USB stick.

(f) Syringe pump

The syringe pump is used for the purging of the solvent lines, for quantitative sample loading and the cleaning of sample vial, needle and sample loop.

(g) Sample loading unit

Quantitative transfer of the sample into the sample loop.

(h) Heated sample loop

Sample reservoir during the process.

(i) Column tower

Patented column tower for clamping the three clean-up columns together.

(j) LED

Status display of the instrument.

- Green: waiting/ready
- Orange: in process
- Red flashing: error/break

(k) Nitrogen valve

Valve unit with nitrogen connection for optional drying of the columns.

(l) Solvent supply

The solvent supply lines are marked with the following abbreviations:

- SP n-Hex: Solvent n-Hexane. Line directly to the syringe pump valve
- n-Hex: Solvent n-Hexane. Line directly to the pump supply valve
- SP Toluene: Solvent Toluene. Line directly to the syringe pump valve
- Toluene: Solvent Toluene. Line directly to the pump supply valve
- SP DCM/n-Hex 1:1: DCM (Dichloromethane)/n-Hexane (50:50) Line directly to the syringe pump valve
- DCM/n-Hex 1:1: DCM (Dichloromethane)/n-Hexane (50:50) Line directly to the pump supply valve
- DCM/n-Hex 2:8 DCM (Dichloromethane)/n-Hexane (20:80) Line directly to the pump supply valve

(m) Control Unit

Unit to adjust the temperature for heated parts.

3.2. Rear Side of the Device

(a) Power supply
Power supply of the system with 36V.

(b) Power supply
Power supply of the system with 24V.

(c) Waste sensor
The waste sensor is used for monitoring the liquid level in the waste bottle. If the waste bottle is
full, the waste sensor interrupts the clean-up process and a message will appear on the screen.

(d) Waste tubing
The waste lines need to be connected with a suitable waste container.

(e) Nitrogen connection (optional)
It is possible to connect a nitrogen supply (¼”-28 UNF thread). If no nitrogen is required, the nitrogen connection must be closed off with the provided adapter.

(f) Horn
Acoustic signal indicating an error, break or the end of the process.

(g) Power supply
Power supply of the heating unit with 24V.

(h) Connections for the heating elements
Electrical interfaces: Channel 1 - Vial, Channel 2 - Sample Loop, Channel 3 - Tube Heating.

4. Commissioning

4.1. Installation

- Connect waste sensors (optional) or dummy plugs.
- Connect to a power supply (100-240 Volt AC, 50-60 Hz).
- Set the pressure for the nitrogen supply connection (optional) to 3 bar ~ 40 psi.
- Set up solvent racks (optional) and connect solvent tubes with the associated solvent bottles (solvent bottles must be at the same level as the device).

    1. Connection: n-Hexane
    2. Connection: Toluene
    3. Connection: DCM (Dichloromethane)/n-Hexane (50:50)
    4. Connection: DCM (Dichloromethane)/n-Hexane (20:80)

    In order to avoid any damage of the pump you are advised to use solvent filters. These have to be cleaned with n-Hexane before use. Furthermore, the pump should not run without any liquid.

- Attach the fraction rack (optional).
- Connect the fraction line to a suitable fraction collection vial.
- Connect waste tubes to waste containers. For uninterrupted waste drainage, waste tubes and waste container should be situated below the device.
- Connect the temperature control unit to the heating parts.

4.2. Checklist Initial Operation

The DEXTech Heat system is now ready for the initialization:

- Control waste bottles and if necessary, empty the waste bottles.
- Fill all solvent reservoirs.
- Switch on the system and choose the set-up.
- Switch on temperature control unit (can take up to 15 minutes to reach the temperature).
- Lock column tower.
- If required, adjust language (English-German, Chapter 5.5.1), pressure unit (bar or psi, see Chapter 5.5.3) and time/date (Chapter 5.5.4).
- If necessary, put in the dummy column and purge the pump, solvent lines and sample loop (Chapter 5.7).

    NOTE: To make sure that the complete tubings are filled with solvents, the respective purging step must be executed twice.

- Rinse the system (see chapter 5.8).

- Rinse the system with “Fraction & Needle” (Chapter 5.8).
- Create or edit the desired method or use the “Alumina Plus”, “Alumina Pure” method or Dioxin only method (see Chapter 5.4).
- Attach the suitable fraction vials to the system or attach the optional fraction rack.

Start the process by inserting the columns (Chapter 5.3.1), select the desired method (Chapter 5.3.2), put in sample and enter user name (Chapter 5.3.3), insert the sample vial (Chapter 5.3.4), and start the process.

ATTENTION!

It is essential to ensure that the operating conditions for the system are kept constant and stable during processing! Otherwise the system could be damaged or the samples may be processed incorrectly.

Please be aware that low boiling solvents (e.g. dichloromethane) cannot be processed, if the ambient temperature is too high!

In addition, the solvent mixture dichloromethane/n-hexane (50:50) and dichloromethane/n-hexane (20:80) should not be used when it has been freshly mixed. It is advisable to let the mixture rest for one day to degas. Failure to do so may lead to inadequately processed samples!

Only samples can be processed, which are suitable for a liquid handling system, which means they are compatible with the demands of the system regarding particles, viscosity, volatility and chemical stability!

Furthermore, the pump should not run without any liquid.

4.2.1 Re-Commissioning after Short-Termed Standstill

Check the solvent and waste container levels.
Check the system for leakage after any transport or disconnection of tubings.

4.2.2 Re-Commissioning after Long-Termed Standstill

After a prolonged standstill, proceed as described under “Initial Operation”.

5. Software

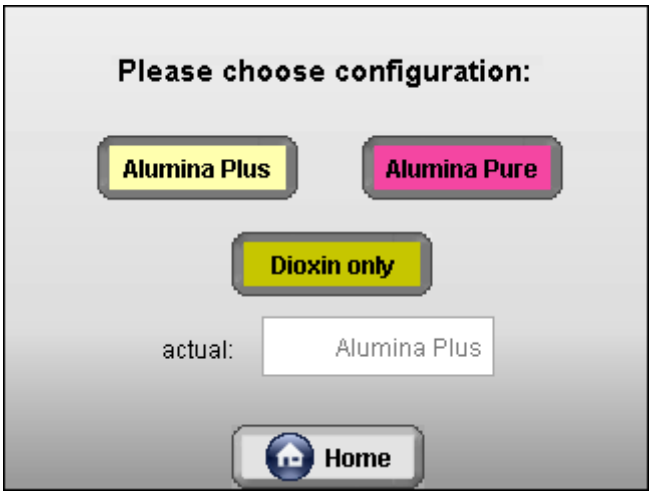
The DEXTech Heat display (touch screen) serves as the control interface for the system, which allows for easy and efficient control by the user.

5.1. Start Screen

The following screen should appear after switching on the system. The initial operation progress is shown on the blue bar in the lower part of the screen.

Figure 1: Start screen after switching on the power supply.

Once the start screen is completed, a request (Figure 2) appears asking which set-up you would like to choose (“Alumina Plus” or “Alumina Pure”). The selection is important for sample processing. Afterwards you can always change this in the main menu.



5.2. Main Menu

In the main window you find different options (Figure 3):

a. Process (Chapter 5.3)
Link to sample process.

b. Create Methods (Chapter 5.4)
Edit/create methods.

c. Settings (Chapter 5.5)
Settings of date, time, pressure, language, display, volume, service, solvent, interface syringe pump and user names.

d. Lock, Password (Chapter 5.6)
Lock/Unlock methods and settings.

e. Purge(Chapter 5.7)
Fill up pump and solvent tubings.

f. Rinse (Chapter 5.8)
Additional rinse process for needle and sample loop.

g. Combine (Chapter 5.9)
Combines the purge process of the piston pump, syringe pump with the rinsing of the sample loop, needle and fraction lines. One Step preparation of the system.

h. Report (Chapter 7)
Transfer of the sample data to the USB stick.

i. Set-up (Chapter 8)
Change configuration between “Alumina Plus”, “Alumina Pure” or “Dioxin only”.

j. Software version

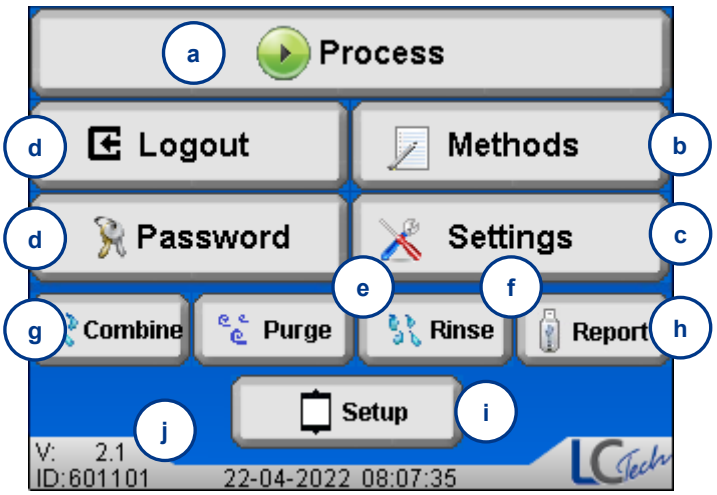


Figure 3: Main window.

5.3. Process

To prepare processing, various steps need to be undertaken, which include for example insertion of the columns into the column tower, selection of the desired method etc. Each step will be explained in the following chapters.

5.3.1 Insertion of the Columns into the Column Tower

The first step in starting the clean-up of a new sample is to insert the three columns into the column tower. This is quite simple. Take out the columns from the storage bags and “just click it!“ into the column tower.

1.a 1.b 1.c

1.a	Universal column for Dioxin analysis Product No.: 15068
1.b	SMART column for Dioxin analysis Product No.: 14307
1.c	Adapter for SMART column Product No.: 14262

2 3

2	Alumina column Product No.: 15433
3	Carbon column Product No.: 15242

Figure 4: Available columns for processing with the respective description and product number.

INFORMATION: Two different column types are available for column position no. 1: the Universal column and the SMART column. The SMART column can be used for samples of up to 1.5 g of fat. The Universal column can be used for samples of up to 5 g of fat. For processing of difficult samples and fats that harden at room temperature it is highly recommended to reduce the sample intake to less than 3 g.

- ➔ The use of the SMART column requires an additional column adapter.
- ➔ If any of the three column positions is not used, a dummy column needs to be used in its place.

Attention when you choose a „Dioxin only“ method a dummy column has to be placed on column position 2.

INFORMATION: Removal of columns from the packaging.

The columns are either separately (Figure 5: 1.a, 1.b, 2) or jointly (Figure 5: 3) heat sealed in foil.

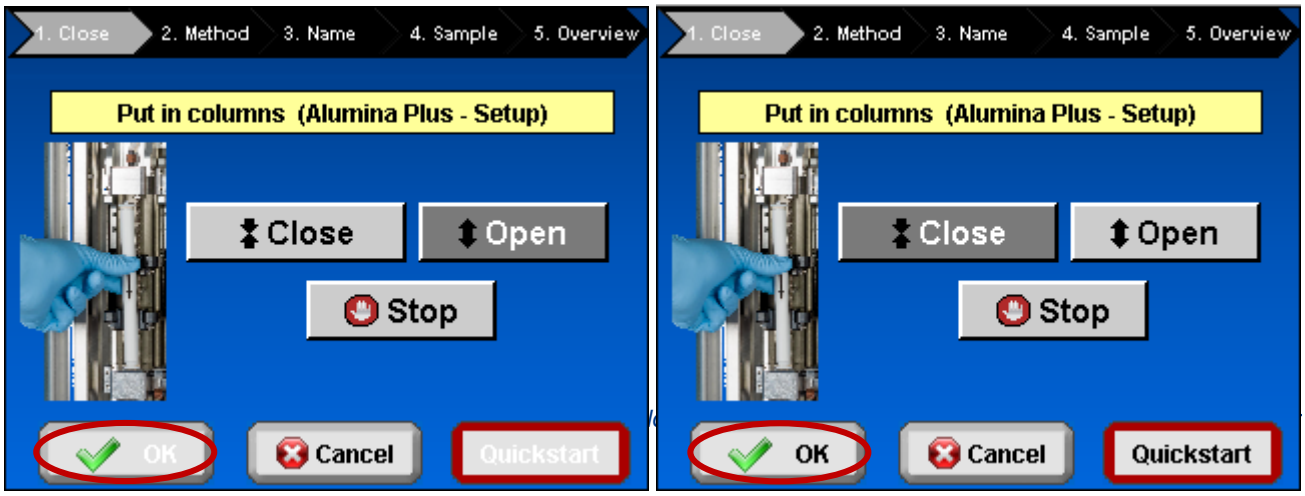
Figure 5: Heat-sealed columns. 1.a Universal column, 1.b SMART column, 2. Alumina column, 3. Carbon column (small).

ATTENTION: The alumina column is available individually sealed in packs of 5 (see Figure 6). Due to deactivation through small amounts of water, it is strongly recommended to remove the columns individually without damaging the adjacent weld seam.

Figure 6: Alumina packing.

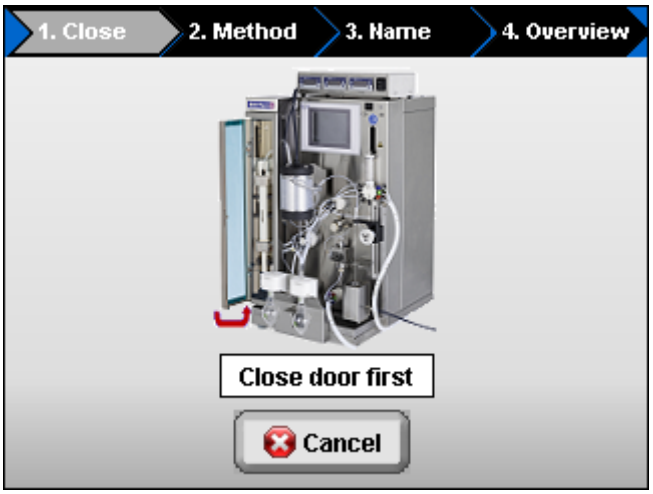
For the insertion of columns, it is important that the column tower is fully open and that each column is fitted in its correct place (clicked in). Otherwise, the system will not close properly.

The column tower is opened and closed via the software. With the *"Open"* button, the column tower opens to its full extent automatically (analogous process applies to the *"Close"* button). Only after closing the column tower (black "*Close*" button), the *"OK"* button will become selectable, enabling to proceed to the next step (see Figure 7, Alumina Plus set-up).



If a problem occurs, the opening and closing of the column tower can be interrupted by pressing the *"Stop"* button.

Safety note: Never put your hand in moving mechanical parts or in gaps intended for mechanical movements! The opening and closing of the column tower may only be undertaken with the front door closed. Otherwise a request to close the door appears, as shown in Figure 8.



See the correct order of columns. This order is fixed to the system and looks always the same.

➔ **The defined column sequence is fixed and may not be changed!**

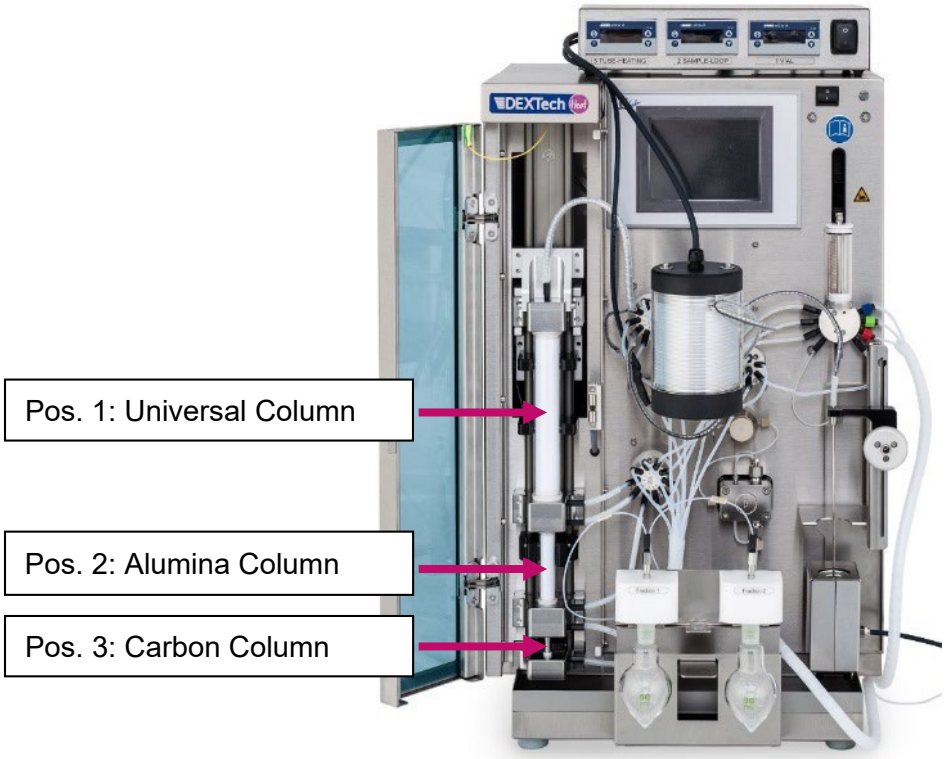


Figure 9: Sequence of the three columns in the system.

For use of the SMART column, an additional adapter is required. The adapter is simply clicked into the system, and then the SMART column can be inserted (see Figure 10).

Figure 10: Handling of the SMART column. First, click the adapter into column position 1 (left) and then insert the SMART column (right).

By pushing the „Quickstart Button “see Figure 7, the system will start the next sample without any intermediary, additional steps. For the sample, the system will choose the same method it has used for the previous sample automatically. As a sample name, the system uses “Quickstart” which will be shown in the report of the sample. There will be no reminder to put in the new columns, sample vial or fraction vial. If the system does not recognize a previous method, the following error message will appear.

Figure 11: Error message for an invalid method.

INFORMATION: After each restart of the instrument or after a change of the configuration, a regular start with selection of the method and name hast to be done. Without this, a Quickstart is not possible.

5.3.2 Select Method

Next step is to select a stored method. You can choose one of the two Default methods (depending on the chosen set-up) or any other stored method on the system. Select a method by pressing the name of the method (a green bar appears with the method name) and then press the *"Select"* button. The name of the method can be seen now in the selected window. Afterwards press the *"OK"* button in order to go to the next step (see Figure 12, Alumina Plus set-up).

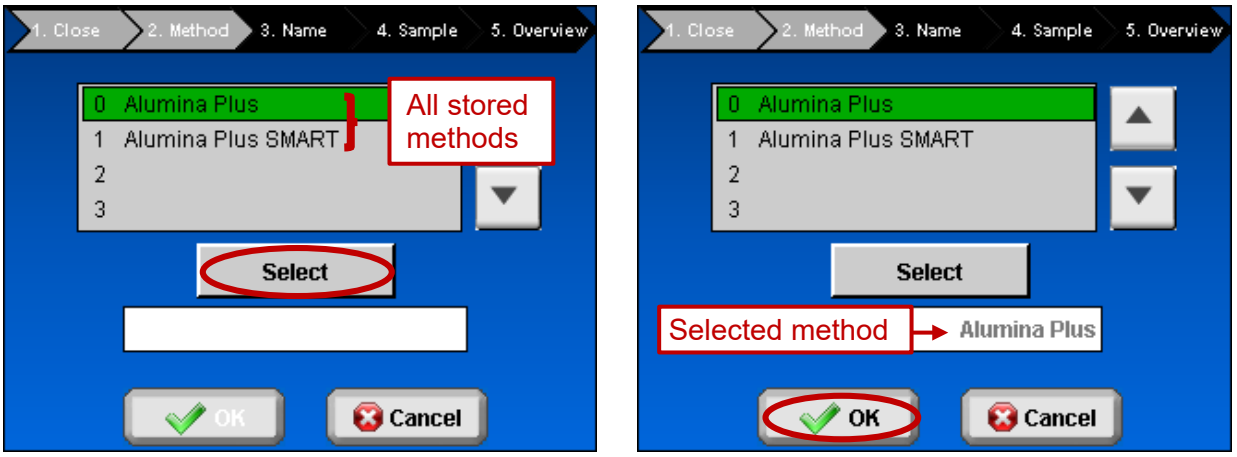


Figure 12: Method selection. After choosing a method (left screenshot) the "OK" button is available (right screenshot).

NOTE: To select a method, click onto the required method. A method is selected once it is highlighted in green.

5.3.3 Sample Name and User

Type in the sample name (without any special characters) and the current user. Upon completion of these inputs, you will be able to proceed to the next step (Figure 13). Pressing the white box automatically opens an input screen in which the desired sample name and user can be entered.

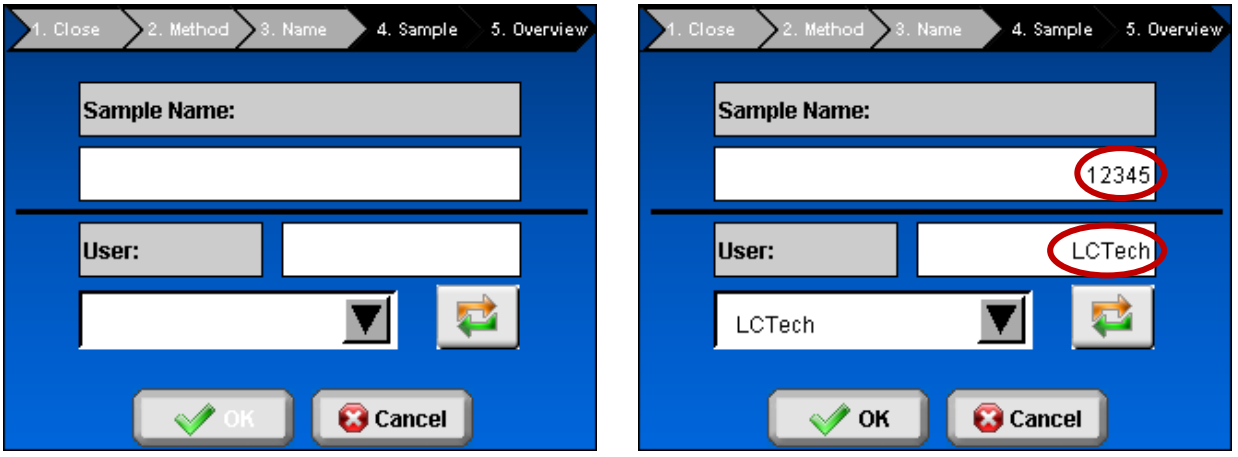


Figure 13: Comparison: Before and after the input.

NOTE: To speed up operation, it is possible to select a user name from the dropdown menu, which has already been saved instead of creating a new user name (see Chapter 5.5.7). Just select the desired name from the list and press the "*arrow*" button. Subsequently, the user name will be inserted in the user field.

5.3.4 Sample

This process window is a reminder to provide the fraction collection vials and to place the sample vial into the sample vial holder. In addition the heating state has to be set (Figure 14).

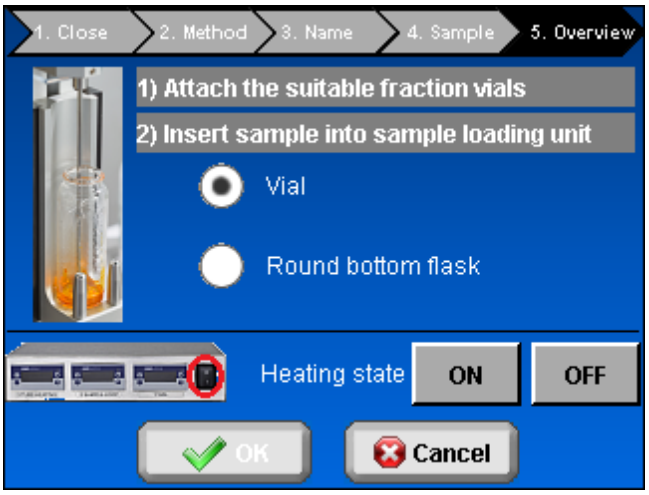


Figure 14: Sample window.

Figure 15 shows the option to choose between a vial and a round bottom flask as sample vials. For the round bottom flasks, a waiting time during the sample vial rinsing process can be chosen. During this waiting time the rinsing solution, which has been sprayed to the glass wall, has time to gather at the bottom of the vial before it is sucked into the sample loop.

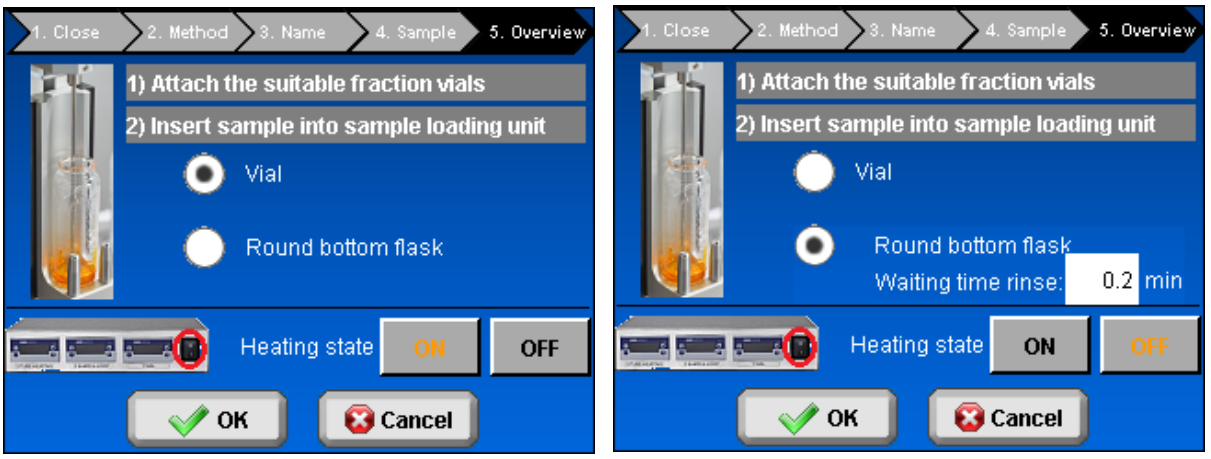


Figure 15: Reminder to provide the sample collection vials and to place the sample vial into the sample vial holder.

Before the process is started, the sample vial must be placed into the sample loading unit. By pressing the push button the holder can be pushed downwards until the needle reaches the bottom of the sample container. The following figure illustrates this procedure.

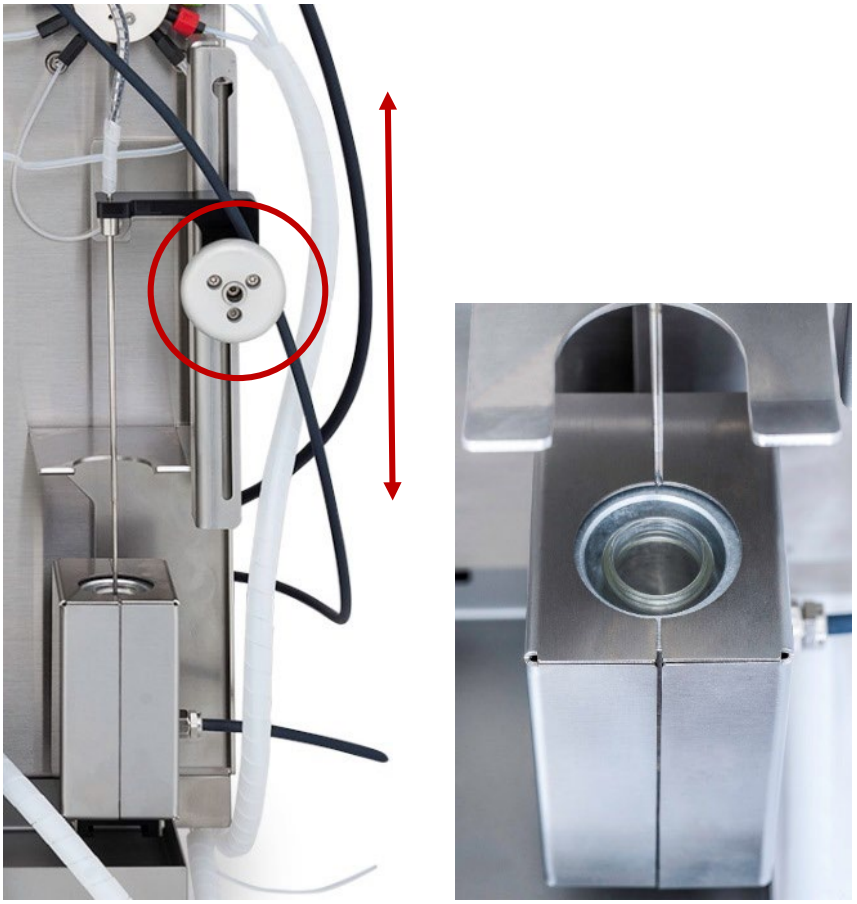


Figure 16: Sample loading unit for placing the sample vial in the sample vial holder

NOTE: The control unit of the DEXTech Heat system has to be switched on separately. (Figure 16).

Figure 17: Temperature control unit.

5.3.5 Overview

The “Overview” window shows all important data (method, sample name, user and duration) of the sample (Figure 18).

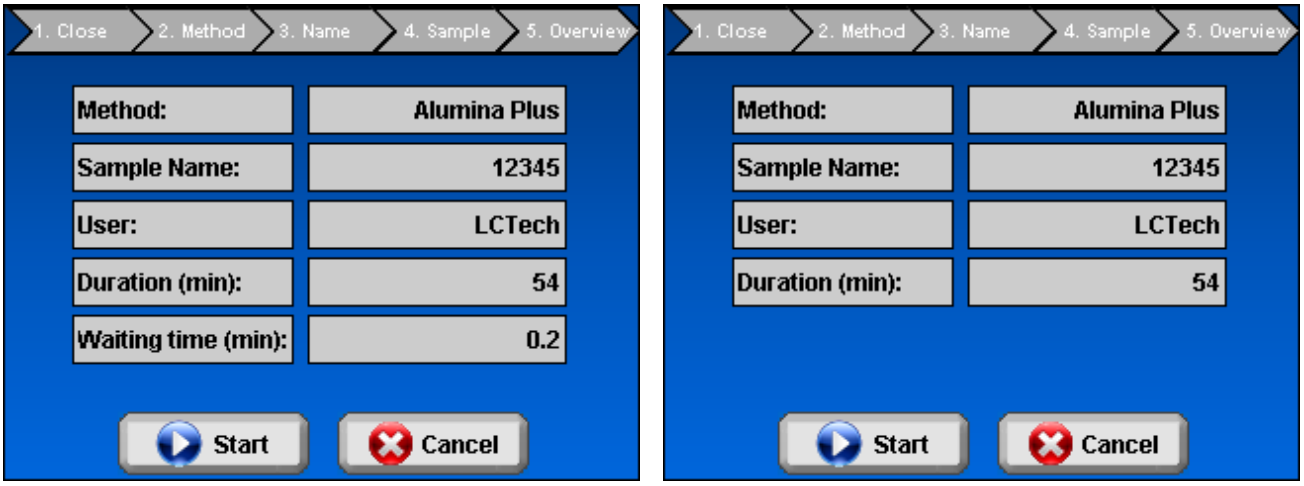


Figure 18: Overview.

INFORMATION: You can jump back to any earlier step by pressing the desired button (Figure 17) with the respective heading.

In the event that the maximum pressure for column 1 has been set above 5.0 bar (72.6 psi), additional information on the set maximum pressure value for the column 1 will appear in the overview window. For example: Note: max. Pressure C1= 72.6 psi

5.3.6 Process

The progress of the method is monitored and visualized on the process screen (see following figure with explanations).

i. Overview

The overview page provides information about the status of the whole test treatment. This page contains the following information:

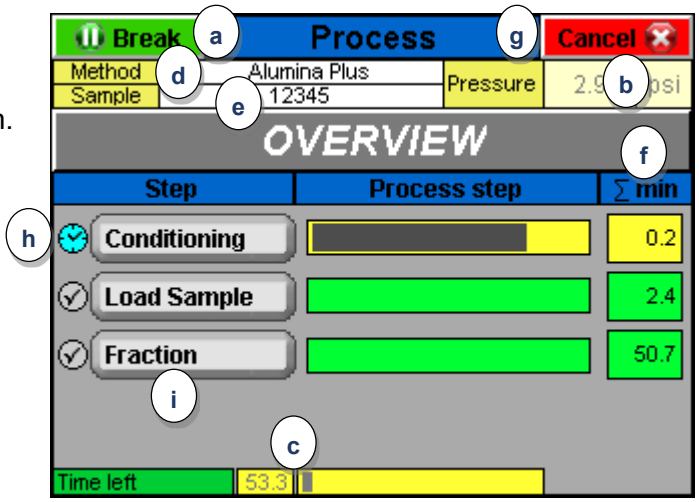


Figure 19: Process screen "Overview" with explanation on the different functions:

(a) Break
Option to stop and resume the process.

(b) Pressure monitoring
Display of actual pressure of the system.

(c) Time left
This is the amount of time left to reach processing end.

(d) Name of method

(e) Name of sample

(f) Time remaining
Announcement of the remaining time for the respective process segment. Values are dependent on the method chosen.

(g) Cancel
Abort the session and continue with rinsing the system.

(h) Status display
Announcement of the current state of the process segment.

- Wait
- Processing
- Finish
- Error

(i) Buttons
The buttons *"Conditioning"*, *"Load Sample"* and *"Fraction"* allow a top view on the respective process segment.

Colour coding of the progress bar:

- Non-activated step
- Already processed step
- Current processing step (the grey bar defines the real time progress)
- Outstanding processing step

ii. Top view

Contains a detailed view of the respective process segment with a listing of the individual steps. These are described in the following:

Conditioning Figure 20 illustrates the top view of the conditioning.

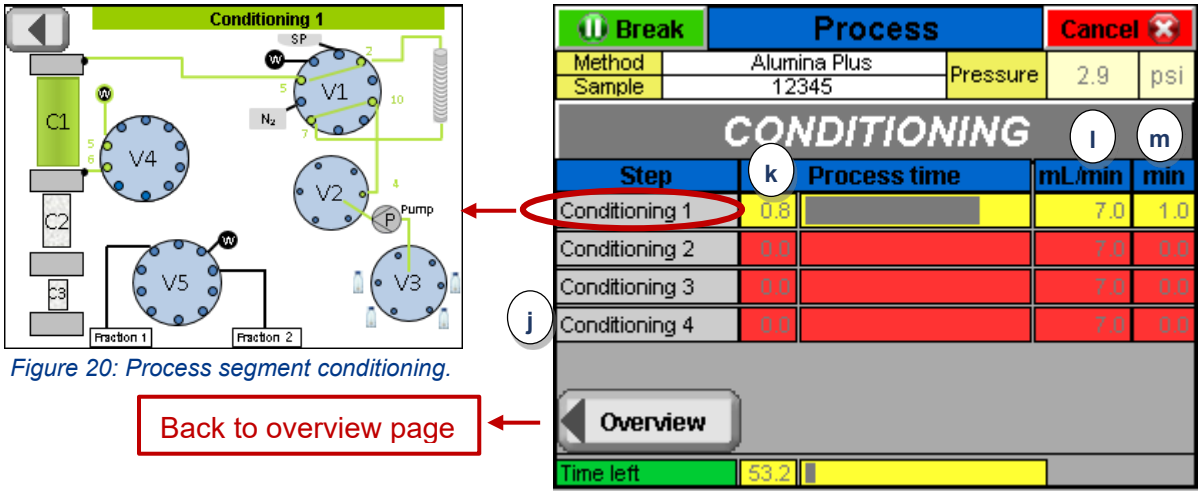


Figure 20: Process segment conditioning.

Back to overview page

(j) Step
List of all conditioning steps.

(k) Actual time

(l) Flow rate
Value of the selected method.

(m) Adjusted time
Value of the selected method.

Load Sample The steps for sample injection and rinse vial are contained under the process point "Load Sample" (Figure 21).

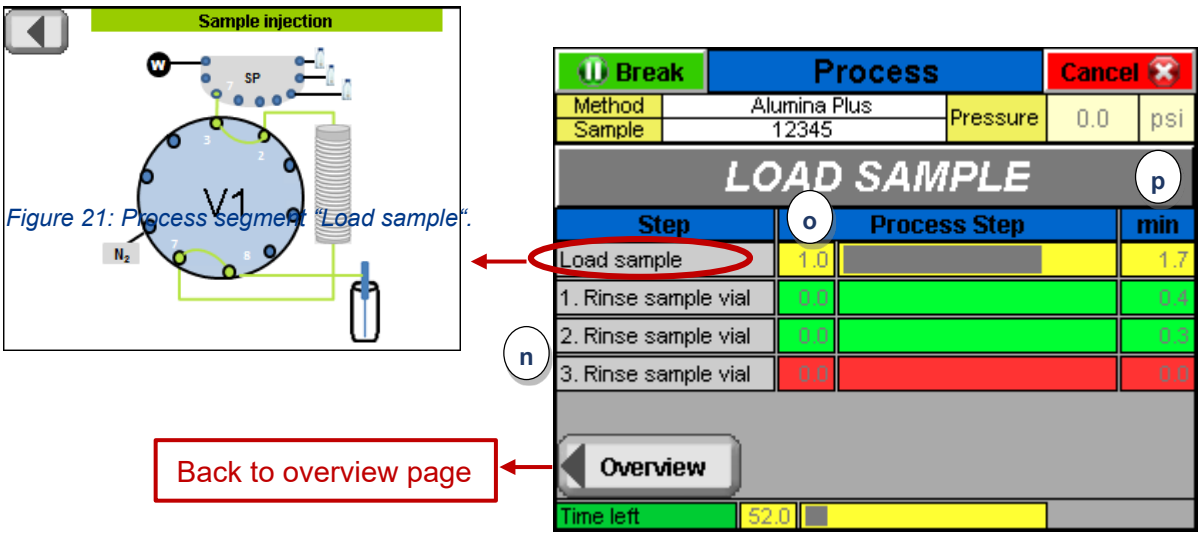


Figure 21: Process segment "Load sample".

Back to overview page

(n) Step
List with all steps.

(o) Actual time

(p) Adjusted time
Value of the selected method.

Fraction

This section (Figure 22) provides pre-run steps and fraction steps.

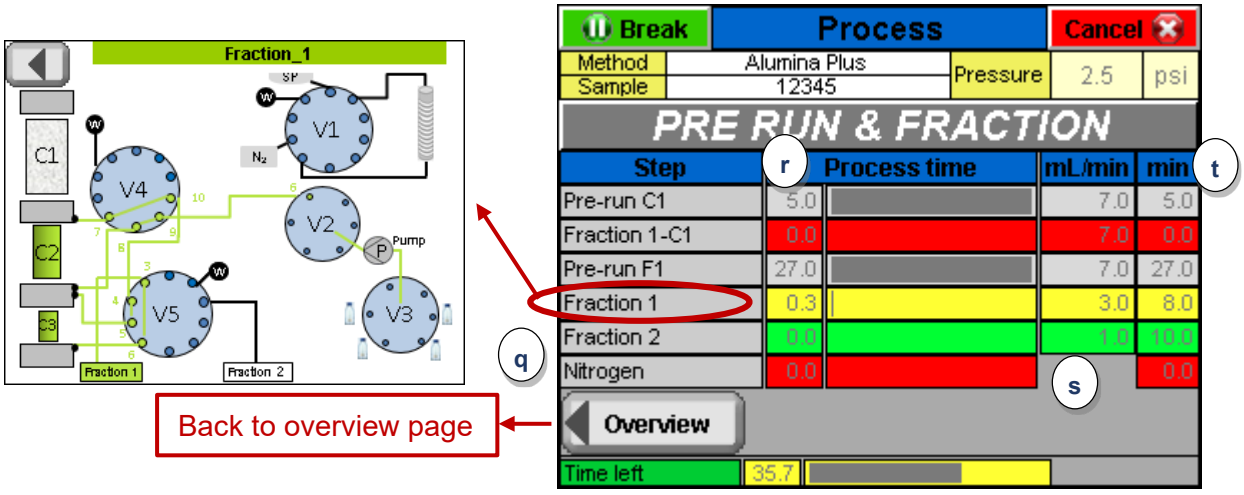


Figure 22: Process segment pre-run and fraction for Alumina Plus set-up.

(q) Step
Arrangement of all possible steps in this sub-range.

(r) Actual time

(s) Flow rate
Value of the selected method.

(t) Adjusted time
Value of the selected method.

5.3.7 Incidents during Processing

I. Break

You can stop the process at any time. The break screen shows the current processing step (see Figure 23). You can resume or cancel the process. In this mode it is possible to open the column fixation, but it is not recommended if there is no problem.

ATTENTION: When opening the column tower, solvent can escape in an uncontrolled manner. Please wear hand protection!

In addition, the “BREAK” window provides information about the current step, the time already passed (during the current step) and stop times (when the process was interrupted).

NOTE: If the device is in BREAK mode, a beeping sound will signal the process interruption. By clicking on the speaker, the sound can be turned off and turned on again. When exiting the *“*BREAK*”* window, the speaker will be automatically turned back on. An additional LED changes colour from red to green.

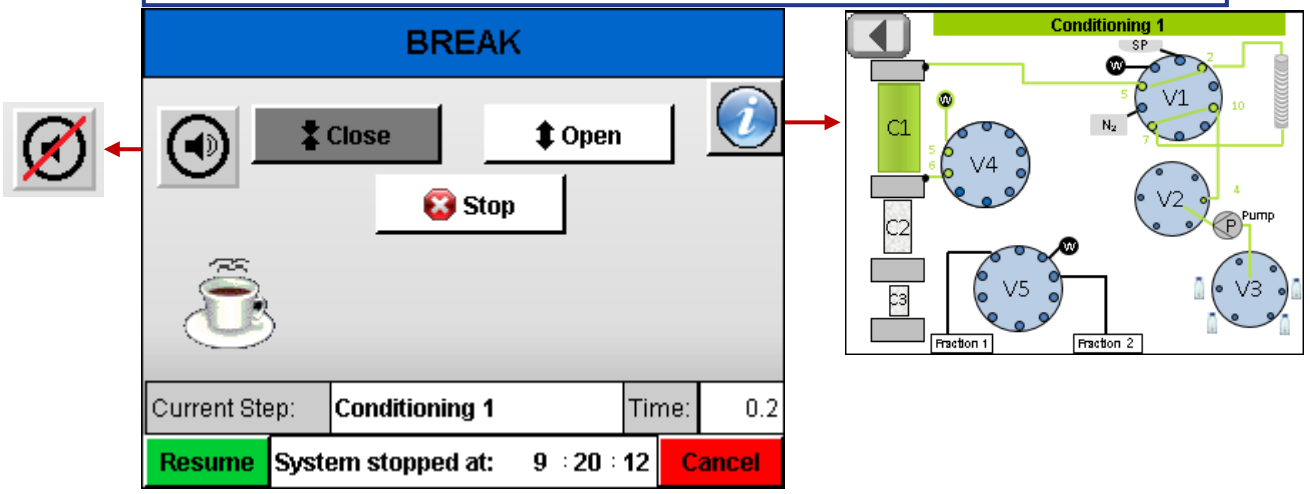


Figure 23: Break window with information on stop time, the current processing step and time passed during this step. In addition, the current flow diagram including the corresponding valve positions can be viewed.

In the event that an error occurs during the process, the system is immediately stopped and put into *“*Break*”* mode. The triggering error will be displayed in the window indicating the current step, the interruption time and the time passed during the current step. In addition, the current flow diagram including the corresponding valve positions can be viewed by pressing the “*i*” button.

For more information about error messages see Chapter 11 Troubleshooting/FAQ (Frequently Asked Questions).

III. Cancel

Select the *"Cancel"* button in the process window. Before the process is terminated, the user will be asked whether the process is to be cancelled (see Figure 24). Confirmation with *"YES"* will stop the sample and *"NO"* will return to the original process and the sample run will continue.

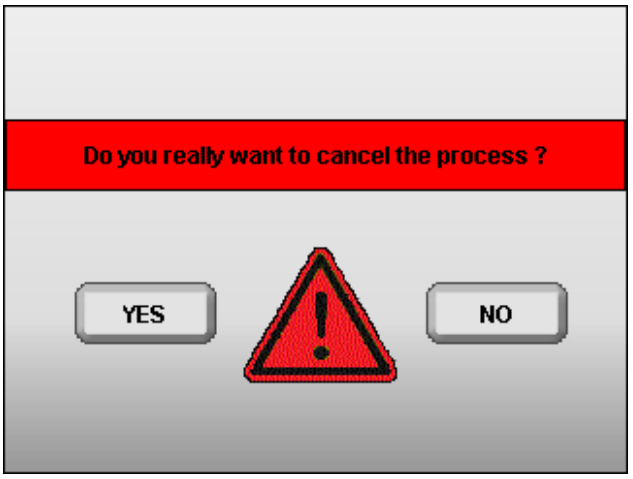


Figure 24: Query window for process cancellation.


ATTENTION: Cancellation of sample processing and subsequent opening of the column tower may cause solvent to escape in an uncontrolled manner. Please use hand protection!

IV. Power failure

After a power cut, a particular window will appear with the return of the power supply (Figure 25, Figure 26). The message depends on the step during which the interruption of the process happened.

If the process was interrupted during sample transfer or rinse vial, an announcement (Figure 25) appears that the process cannot be continued. The user must cancel the entire process. For more detailed information, please refer to Chapter 11.2.13 Power Failure .

Figure 25: Restart window during sample preparation, step sample injection.

In Figure 26, the resumption window shows the option of process continuation. Continuation is possible in the process segments "*Conditioning*" and "*Fractioning*". By pressing the *“YES”* button, the process will continue with the same step during which the power failure occurred. The *"NO"* button indicates that the pre-existing sample preparation is to be cancelled and that rinsing of the system shall be initiated next.

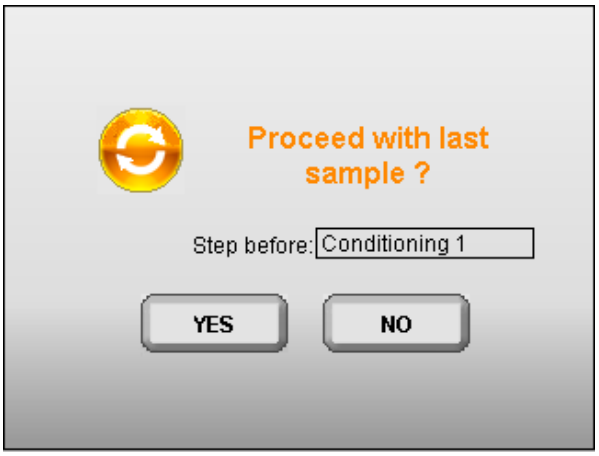


Figure 26: Display screen after power failure.

V. Finish

An acoustic signal indicates when a sample is finished, and the “FINISHED” window will appear (Figure 27). Confirm the window with *”OK”* and the system will proceed with the rinsing step (see Chapter 5.8).

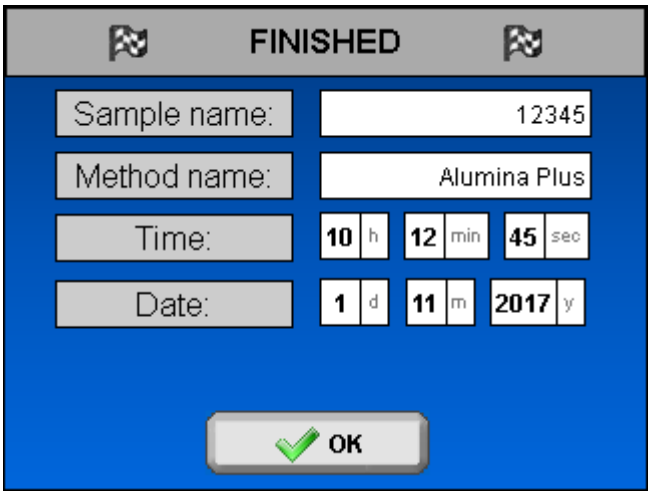
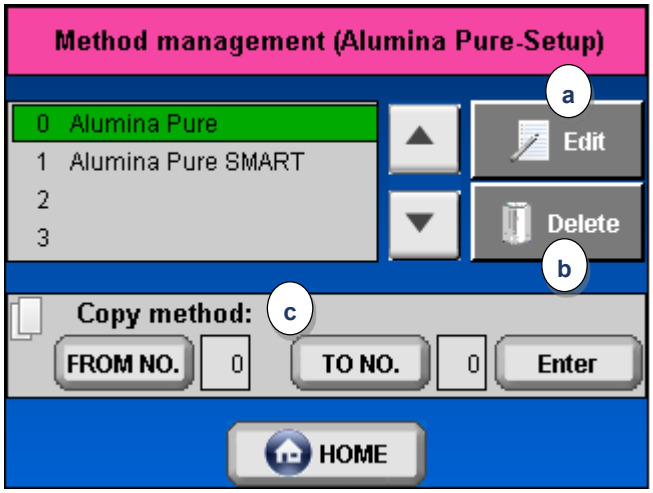


Figure 27: Finish screen.

5.4. Menu Method

This chapter explains the method handling. DEXTech Heat is delivered with six default methods already stored in the software: “Alumina Plus” and “Alumina Plus SMART” methods for the Alumina Plus set-up, “Alumina Pure” and “Alumina Pure SMART” methods for the Alumina Pure set-up (Figure 28) and “Dioxin only” and “Dioxin only SMART” methods for the Dioxin only set-up. The SMART methods are designed for the use of the SMART column (position 1, capacity up to 1.5 g fat), the other two methods for the use of the Universal column (position 1, capacity up to 5 g fat). The following explanations are based upon the Alumina Pure set-up.

NOTE: The methods “Alumina Plus“, “Alumina Plus SMART“, “Alumina Pure“, “Alumina Pure SMART“, “Dioxin only“ or “Dioxin only SMART“ cannot be changed or deleted. For changing these methods, they need to be copied first. Afterwards, a parameter change is possible.



(a) Edit/Create **(5.4.1)**

(b) Delete **(5.4.2)**

(c) Copy **(5.4.3)**

Figure 28: Method management Alumina Pure set-up.

NOTE: A method is only selected, if its name has a green background (see Figure 27).

ATTENTION: when you choose a „Dioxin only“ method a dummy column has to be placed on column position 2.

5.4.1 Editing

You can store up to 30 different methods.

In this mode (Figure 29) you can set-up a new or change an existing method. It is possible to change volume, flow rate and the time for every single step (depending on the respective step). There are options for "*Load sample*", "*Rinse sample vial*", "*Conditioning*" and "*Fraction*". The sample volume can be entered under "*Load sample*". Rinsing of the sample vial can be set to one up to three times under "*Rinse sample vial*". "*Conditioning*" handles all steps before sample processing. *"Fraction"* serves separating fractions.

Pressing the input box allows to name the method or change an already existing one. To confirm the newly created method, press the *"save"* button.

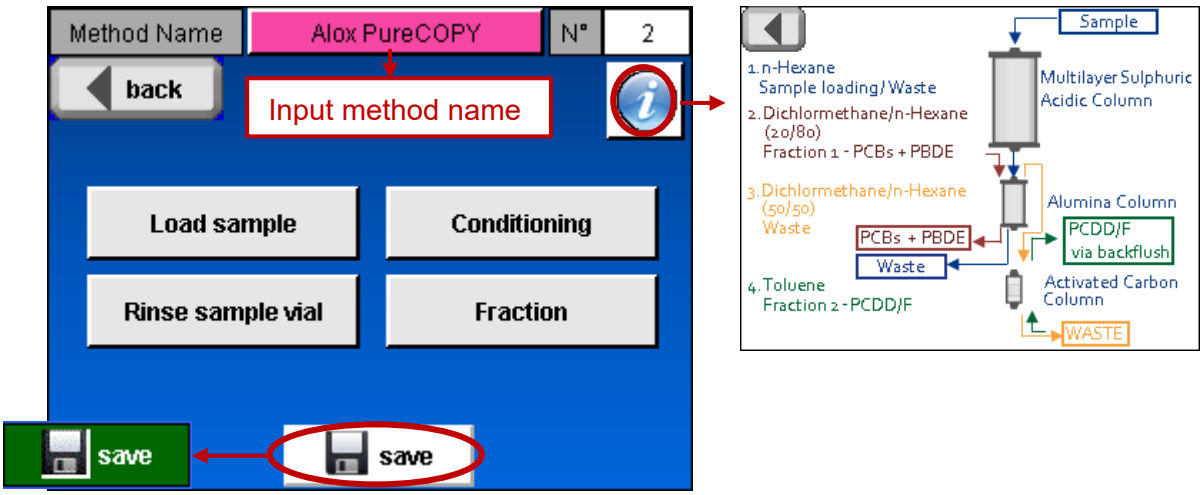


Figure 29: Method editing: input name, information with overview of the process, different steps within the method, and "save" button.

In the event that a method was changed and the memory button was not pressed an additional query occurs asking if the method should be saved (see Figure 30).

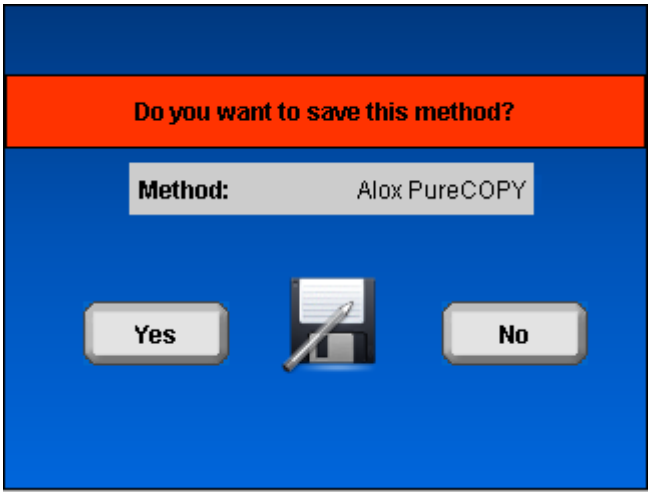


Figure 30: Request to save method.

The individual method steps are described in the following.

Input window for sample volume, represented in Figure 31, which is loaded into the sample loop during the process.

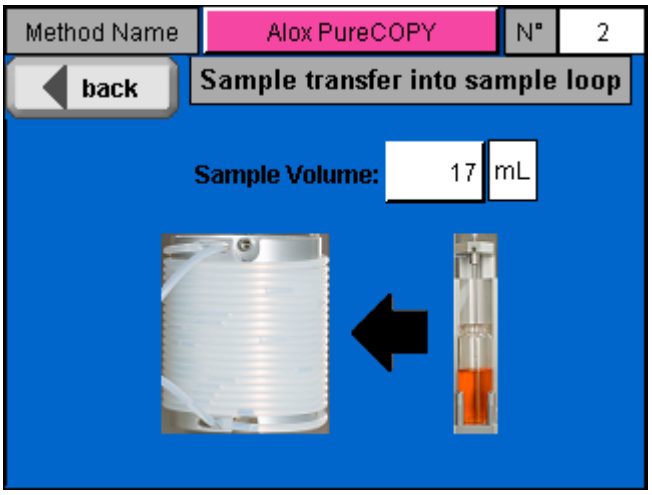


Figure 31: Method step "Load sample".

INFORMATION: The sample volume in the sample vial should be smaller than the entered sample volume in the method to ensure correct function.

For example: Entered sample volume = 17 mL → 16 mL in the sample vial.

Figure 32 shows a warning window that opens when the entered sample volume exceeds the defined maximum sample volume, which is dependent on the sample loop. This window cannot be exited unless the sample volume has been adjusted.

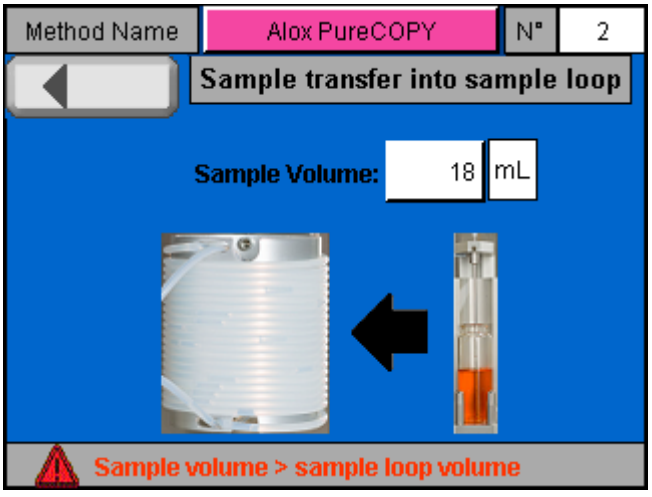


Figure 32: Warning: sample volume exceeds sample loop capacity.

Rinse sample vial

The sample vial can be rinsed up to three times with n-hexane to transfer the whole sample into the sample loop (Figure 33).

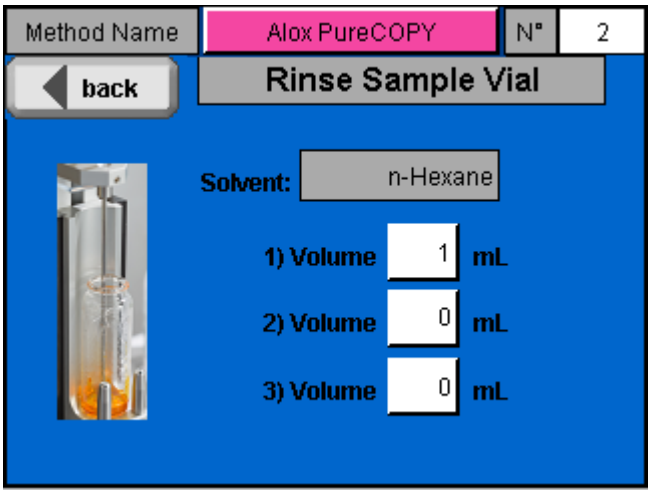


Figure 33: Parameter setting for rinsing sample vial.

Again, a warning window will open if the entered sample volume exceeds the defined maximum sample volume, which is dependent on the sample loop. This window cannot be exited unless the sample volume has been adjusted (Figure 34).

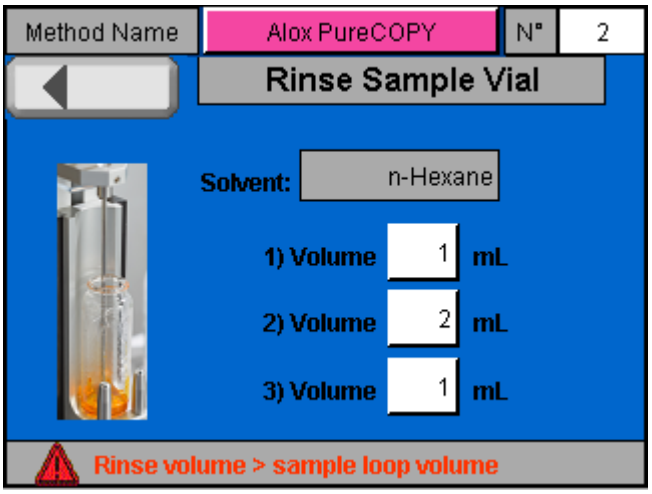


Figure 34: Rinse volume exceeds sample loop capacity.

Conditioning | Fraction

If you wish to change conditioning or fractioning parameters, one of the following screens (depending on set-up) will be shown (Figure 35). Now it is possible to set new parameters for time or flow rate. To change time or flow rate press the respective white boxes.

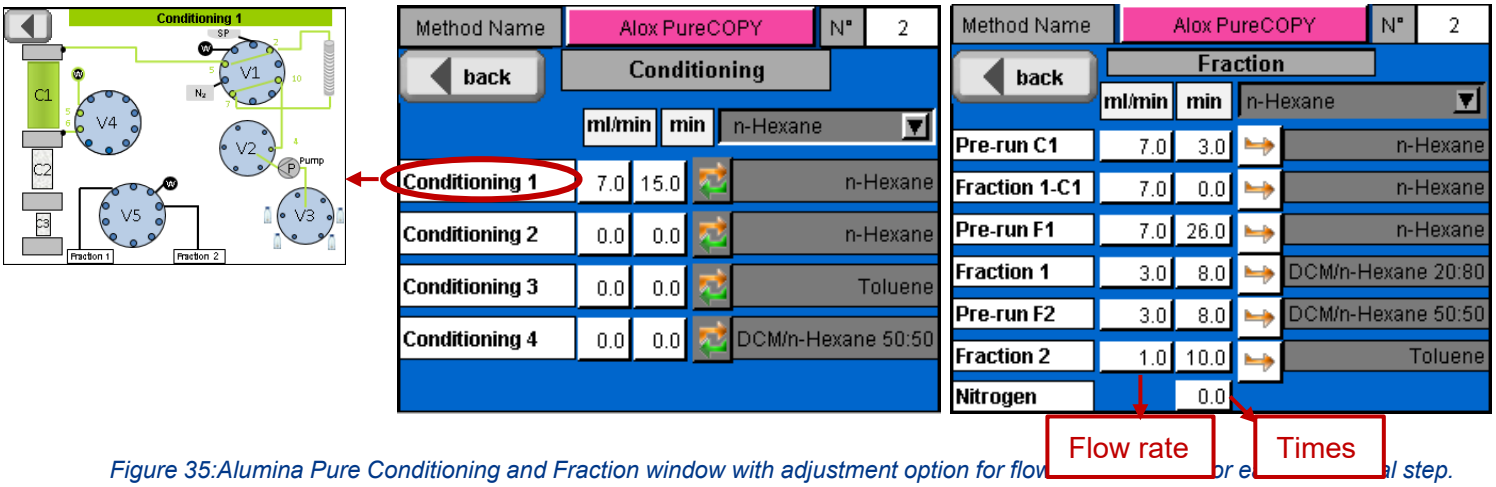


Figure 35:Alumina Pure Conditioning and Fraction window with adjustment option for flow r e l step. In addition, the current flow diagram including the corresponding valve positions can be viewed by selecting the relevant step.

Tip: Do not change the flow rate to values higher than 7 mL/min.

The option to change the solvent in each step is only available after activation of this feature with a level 2 password. If the option is activated, the solvent can be changed in each step by using the thick black arrow and afterwards the double arrow (Figure 36).

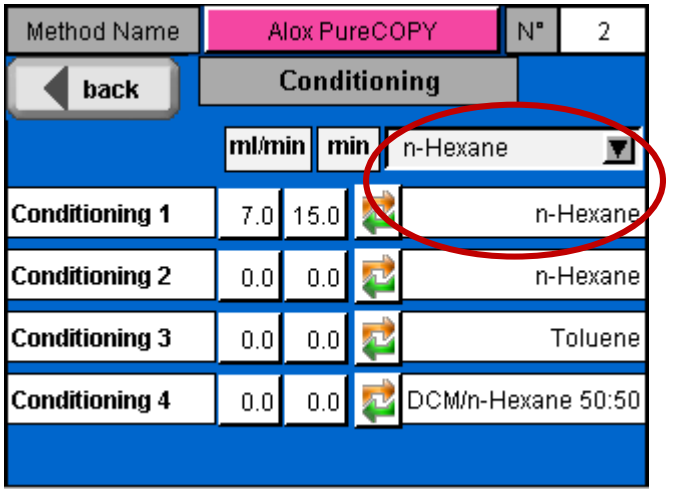


Figure 36: Conditioning with choice of solvent.

Tip: A change of the solvent may significantly influence the method and recovery rates.

Possible sequence of steps within the methods:

Conditioning ➔ Alumina Plus & Alumina Pure & Dioxin only Set-up

Step	Explanation
Conditioning 1:	Conditioning column 1 with n-Hexane.
Conditioning 2:	Conditioning column 1 and 2 with n-Hexane.
Conditioning 3:	Conditioning column 3 with toluene.
Conditioning 4:	Conditioning column 3 with DCM/n-Hexane (50:50).

Fraction ➔ Alumina Plus Set-up

Step	Explanation
Pre-run C1:	Pre-run column 1 with n-Hexane.
Fraction 1 – C1	Fraction1 over column 1 with n-Hexane. (only used for special applications)
Pre-run Fraction 1:	Pre-run column 1 and 2 with n-Hexane.
Fraction 1:	Fraction of mono-ortho PCBs, ndl PCB and PBDE, column 2 (backflush) and column 3 with DCM/n-Hexane (50:50).
Fraction 2:	Fraction of non-ortho PCBs and PCDD/F, column 3 (backflush) with toluene.
Nitrogen:	Drying column 1, 2 and 3 with nitrogen. Delay function for pressure decrease.

Fraction ➔ Alumina Pure Set-up

Step	Explanation
Pre-run C1:	Pre-run column 1 with n-Hexane.
Fraction 1 – C1	Fraction 1 over column 1 with n-Hexane. (only used for special applications)
Pre-run Fraction 1:	Pre-run column 1 and 2 with n-Hexane.
Fraction 1:	Fraction of mono-ortho PCBs, ndl PCB, non-ortho PCBs and PBDE, column 2 with DCM/n-Hexane (20:80).
Pre-run Fraction 2:	Pre run column 2 (backflush) and column 3 with DCM/n-Hexane (50:50).
Fraction 2:	Fraction of PCDD/F, column 3 (backflush) with toluene.
Nitrogen:	Drying column 1, 2 and 3 with nitrogen. Delay function for pressure decrease.

Fraction ➔ Dioxin only Set-up

Step	Explanation
Pre-run C1:	Pre-run column 1 with n-hexane.
Pre-run F2:	Pre-run column 1 and column 3 with n-hexane.
Fraction 2:	Fraction of PCDD/F, column 3 (backflush) with toluene.
Nitrogen:	Drying column 1, 2 and 3 with nitrogen. Delay function for pressure decrease.

For further information, the exact flow diagram is included in Chapter 9 Fluidics.

5.4.2 Delete

If more space is needed or you want to delete a method, select the respective method and press delete (see Figure 28, b). Confirm deletion of the chosen method by pressing the *"Yes"* button (Figure 37).

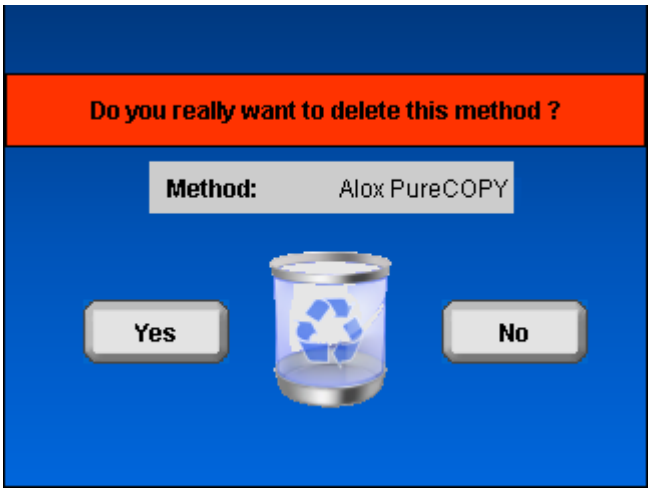


Figure 37: Confirmation prompts for method deletion.

5.4.3 Copy Existing Method

Please follow the sequence of steps described below for copying a method. The copied method will automatically be named with the word "COPY" at the end.

> How to change the name is explained in Chapter 5.4.1 Edit.

Order:

1. Select the method you want to copy – Figure 38.

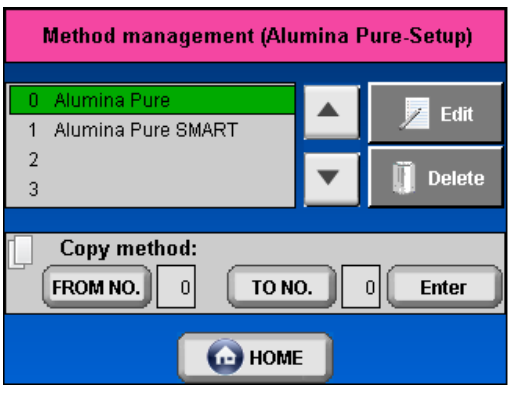


Figure 38: Selected method (green).

2. Press *“FROM NO.”* button – Figure 39.

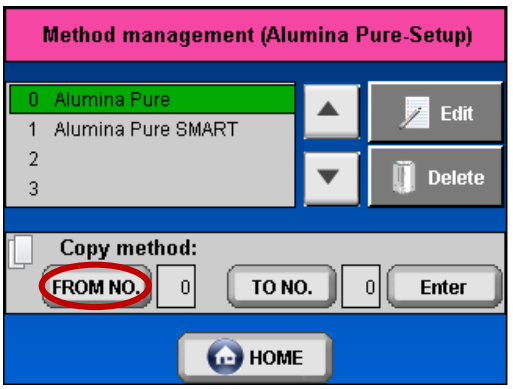


Figure 39: From location.

3. Select a free space in the dropdown menu (max. 30 methods). – Figure 40.

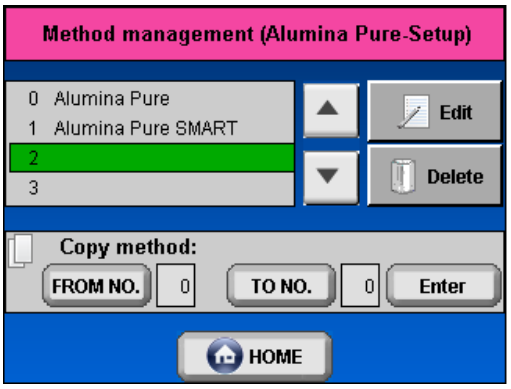


Figure 40: Determine destination.

4. Press *“TO NO.”* button – Figure 41.

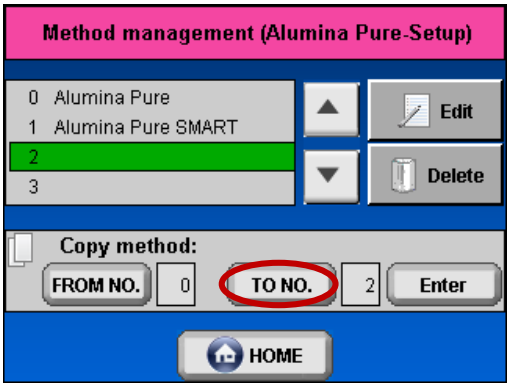


Figure 41: Select “TO NO.” for destination.

5. Confirm with *“Enter”* – Figure 42.

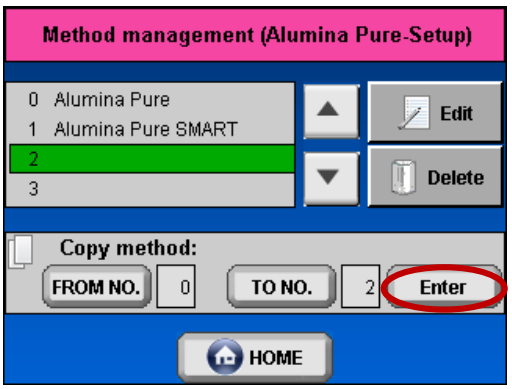


Figure 42: Confirmation.

If the copy process was successful, the new entry will be denoted with “COPY” at the end of the method name (Figure 43, example „Alumina PureCOPY“).

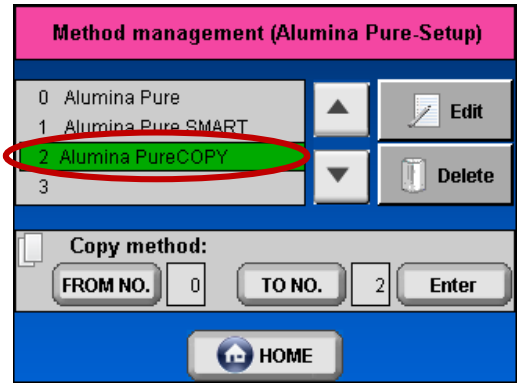


Figure 43: Method list with copied method "Alumina PureCOPY" at list number 2.

NOTE: In the event that the original storage location is identical with the target location, the following message will be shown on the display.

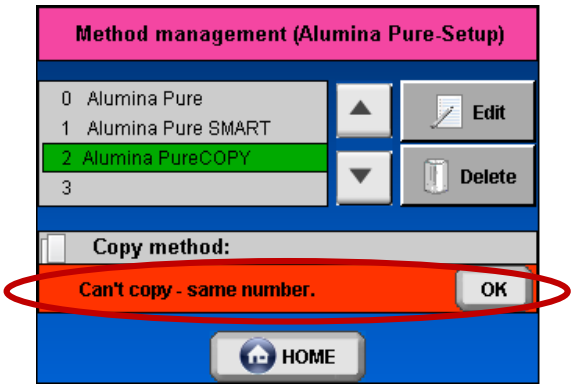


Figure 44: Error method cannot be copied. Selected method “Alumina PureCOPY” should by copied from number 2 to number 2.

ATTENTION: If you copy a method to an already occupied location, this method will be automatically deleted and replaced by the copied method.

5.5. Settings

On this page (Figure 45) different important system and user parameters can be set.

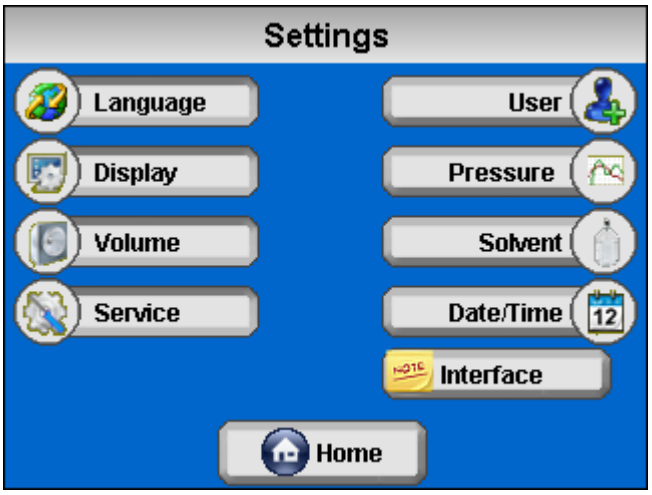


Figure 45: Settings.

5.5.1 Language

Select your preferred language by pressing the language button (Figure 46). The language change takes effect after leaving this window (press "*back*" button).

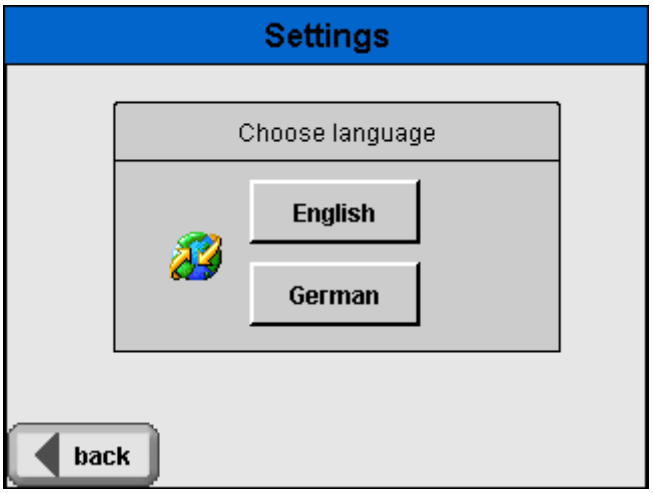


Figure 46: Available languages.

5.5.2 Display

Display settings allow to change brightness and contrast, and to calibrate the touch panel if required (Figure 47). By pressing the "*back*" button, the set values will become effective.

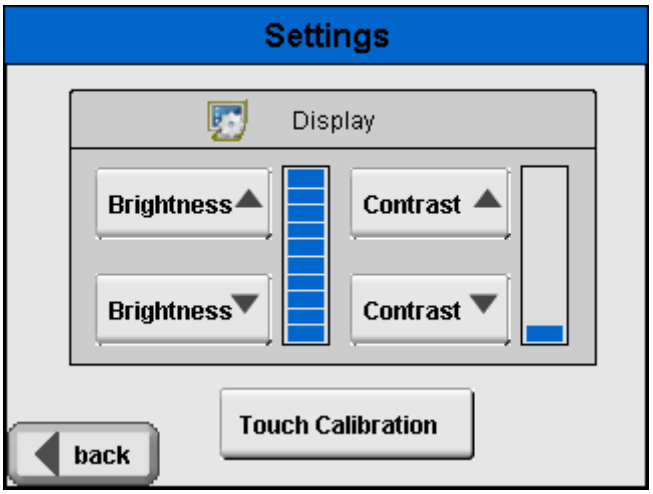


Figure 47: Display settings.

5.5.3 Pressure

The pressure unit of the system can be selected in bar or psi. The selected unit will be displayed in green and the inactivated unit appears in red (Figure 48): The general pressure for the system can be set to a maximum of 15 bar (217.7 psi). In addition, an indicator of the pre-set maximum pressure for column 1 is provided. Both maximum pressure values are set for safety reasons and the system will switch off, if the pressure rises above either maximum set value.

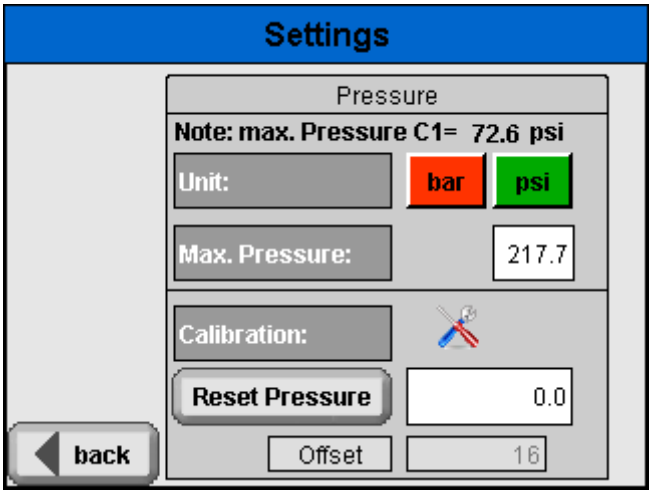


Figure 48: Pressure settings page with the option to choose the unit and to calibrate the pressure sensor.

For installation, it is important to calibrate the pressure sensor (Figure 49). Open the front screw of the pressure sensor and select *"Reset Pressure"* (Figure 48).

ATTENTION: If you wish to calibrate the pressure sensor, the purge knob needs to be opened first. Solvent might drain out, please wear protective clothing.

Executing the calibration:

- Open the purge knob to ensure the system is not under pressure; turn two rotations anti-clockwise. Please use a drip tray to collect solvent that may escape from the valve.
- Click *"Reset Pressure"*.
- Close the purge valve (clockwise).
- The determined value is now stored until the next calibration.

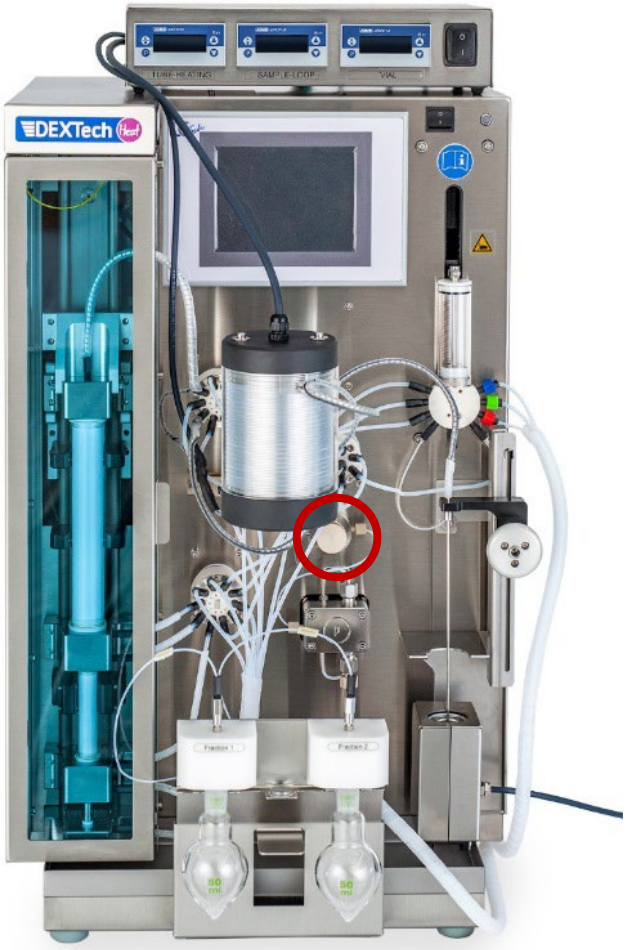


Figure 49: Calibration of the pressure sensor.

5.5.4 Date and Time

Figure 50 shows how to set date and time. Correct settings are important to determine date and time in the event of an occurred error during sample run.

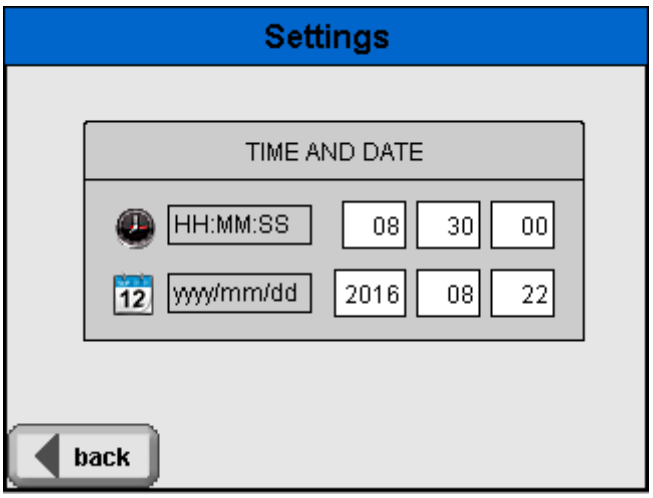


Figure 50: Date and time settings.

5.5.5 Volume

The volume control of the signal tones is shown in Figure 51.

When does the signal appear?

- In the event of a fault (e.g. overpressure, waste full...).
- Processing stops (break).
- At the end of sample run.

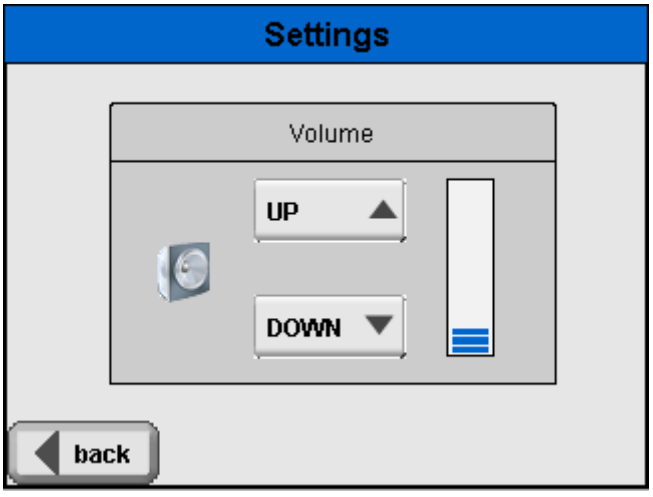


Figure 51: Volume settings.

5.5.6 User

Up to five users can be stored on the system. Pressing the white box automatically opens an input screen in which the desired name can be entered. Confirming with *"back"* saves the data (Figure 52).

This allows for a shortened input during the sample set-up – see Chapter 5.3.3).

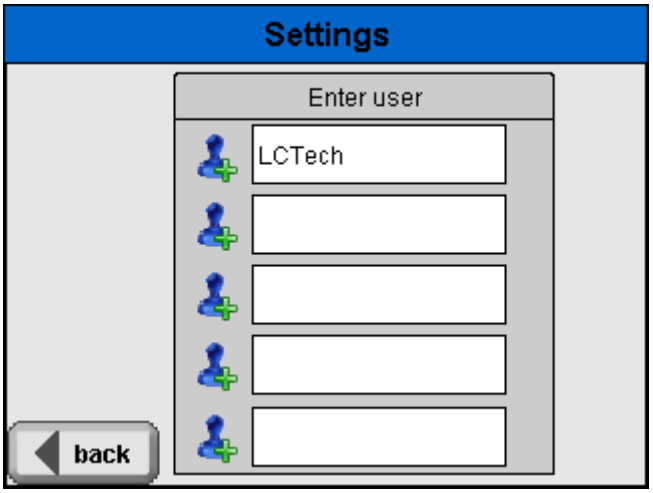


Figure 52: Overview users.

5.5.7 Solvent

Solvent adjustment options: To change the solvents a level 2 password is needed. After the entry of the password, the solvents can be changed.

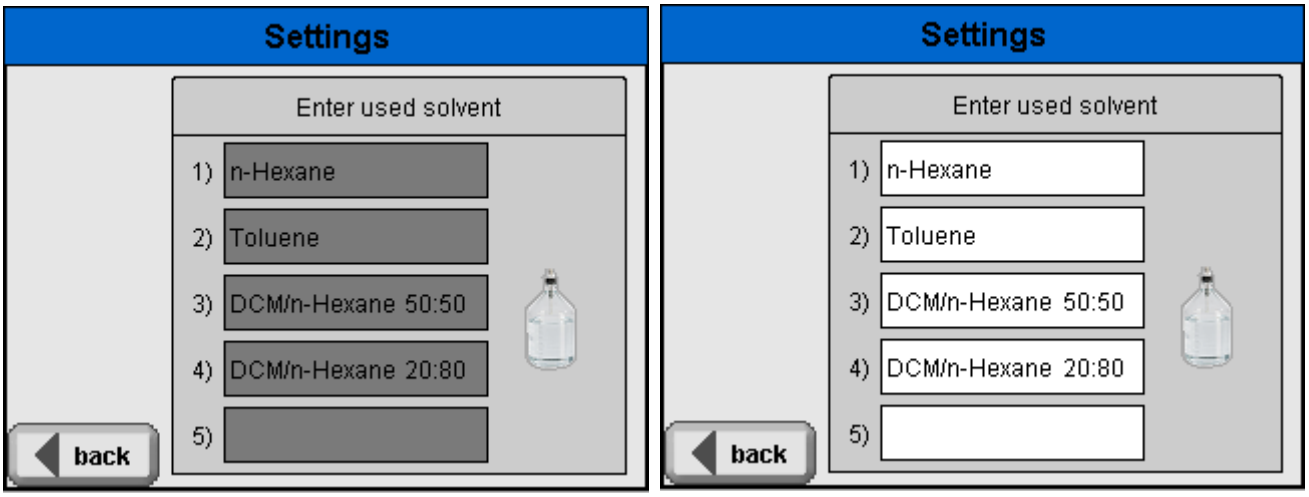


Figure 53: Solvent adjustment on the left: write-protected (without password entry) and on the right: accessible after entry of the password.

5.5.8 Service

In the status display, the status of relevant parts is shown (Figure 54). In the case of an error in one of these parts, a red LED with a related ID number will appear behind the respective part.

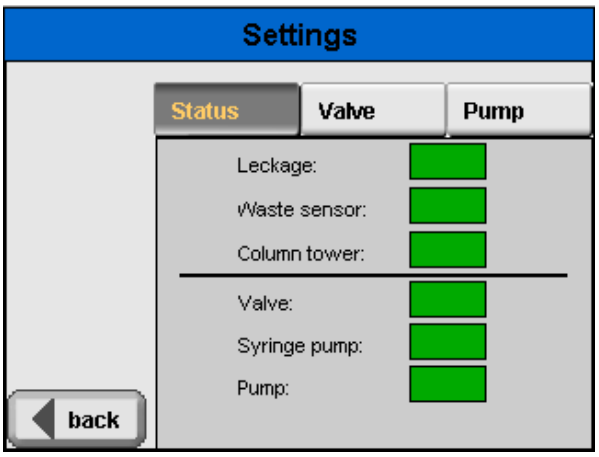


Figure 54: Status display.

The service area contains maintenance recommendations for the rotor seals of the five valves (Figure 55, left) and the wearing parts of the pump (Figure 55, right).

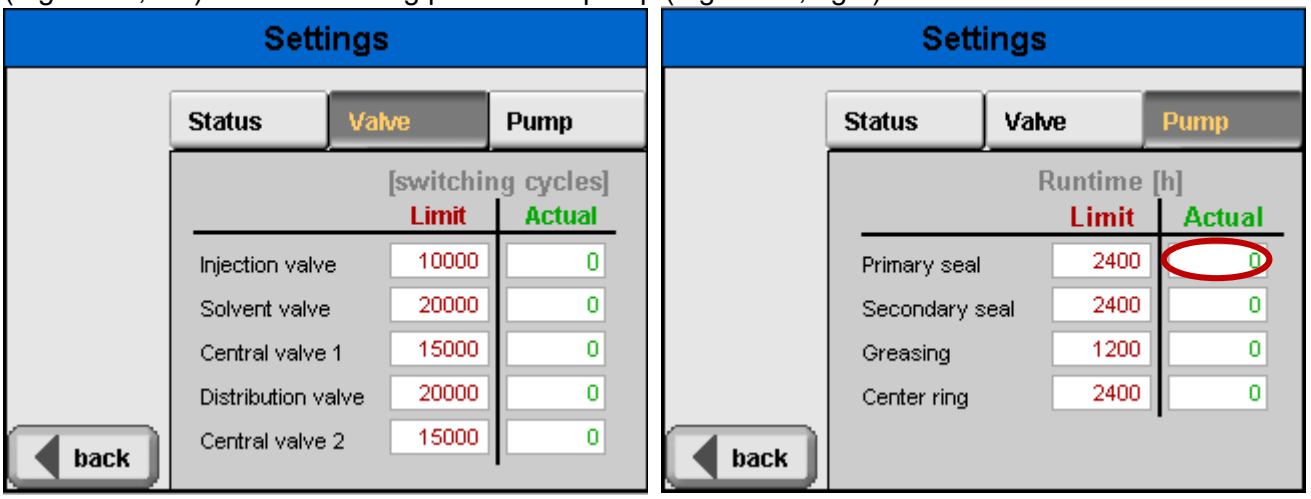


Figure 55: Settings page for maintenance of valves (left) and pump (right).

The *"Limit"* value corresponds to the recommended value at which a component should be replaced. The "Actual" value is the current measured value of the component. Shown values depict the number of performed valve switches or the number of the pump's operating hours.

NOTE: In order to generally deactivate this service function, the "Limit" value must be set to 0. Thus, no service message will appear in the main window. The "Actual" value will still be counted.

Servicing is required when the "Actual" value is exceeded. That is, the "Actual" value is greater than the "Limit" value.

➔ For more information see Chapter 10.3 Software Maintenance.

5.5.9 Interface

Figure 56 shows the data logger of the syringe pump commands.

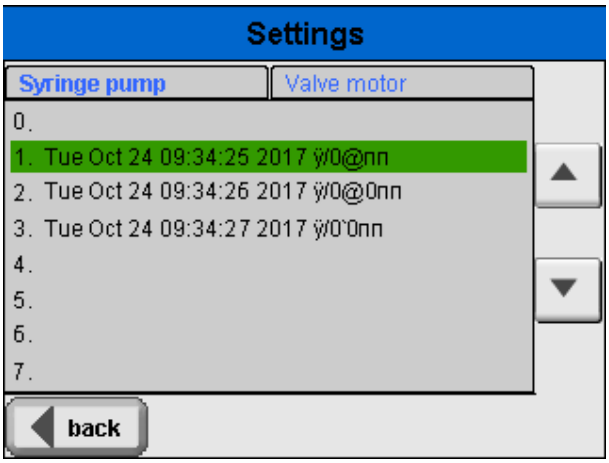


Figure 56: Command list of the syringe pump.

5.6. Password Area

5.6.1 Level 1

The password-protected area level 1 includes the button method management and settings (Figure 57, left). Only after entering the relevant password, the buttons will be unlocked (Figure 57, right).

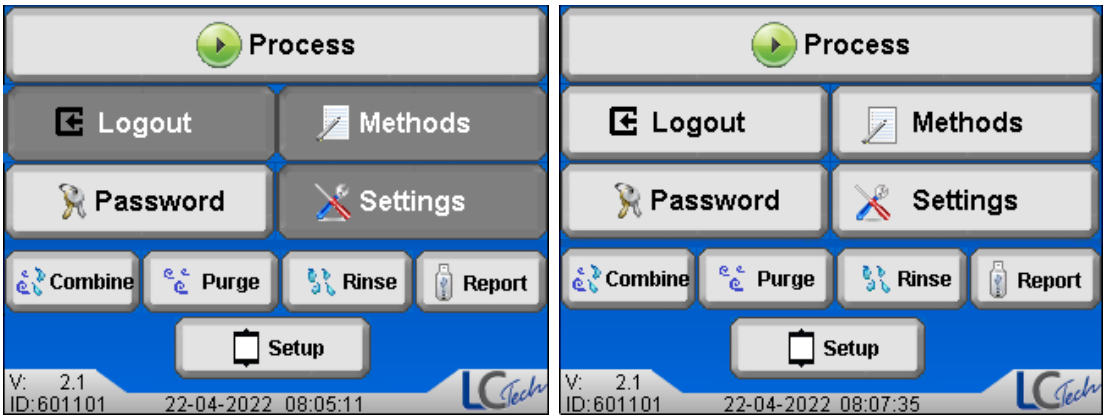


Figure 57: Left screen, locked buttons. Right screen, unlocked buttons "Create Methods" and "Settings".

INFORMATION: The default password for unlocking the creating methods and settings section is set to "**1**". To make sure that the area is further protected, the password of the level 1 should be altered (as described in the following).

To change the existing password press *"Change"* (Figure 58, left). Confirm your old password, type in your new one and press *"Apply"* (Figure 58, right).

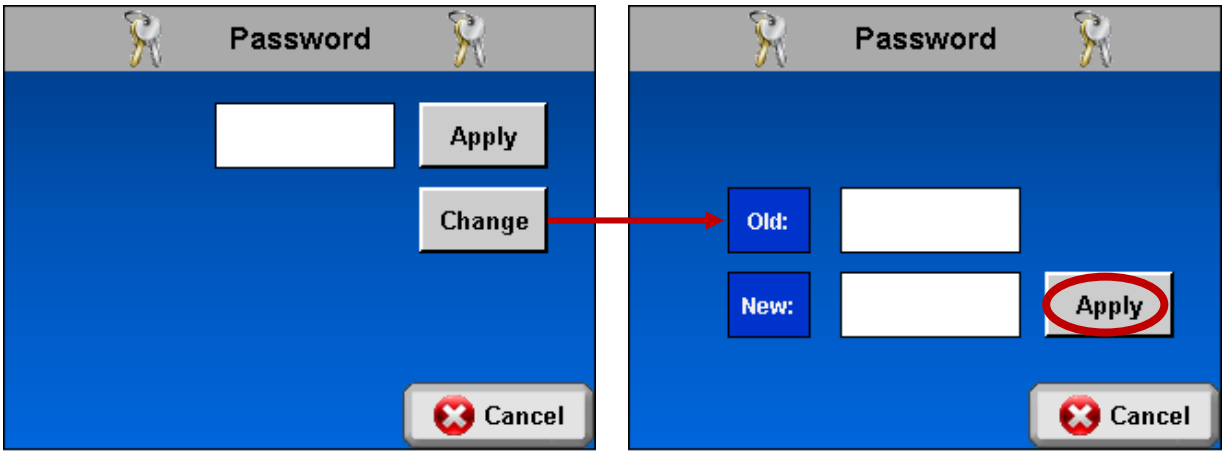


Figure 58: Change password.

5.6.2 Level 2

The internal area of the software (Chapter 10.3.1) is protected by the level 2 password. The access password is available on enquiry through LCTech and cannot be altered.

NOTE: If the wrong password is entered, the following error message will appear on the display.

Figure 59: Error message for wrong password.

5.7. Purge

The system has to be purged to enable fill-up of the solvent lines and to remove potential bubbles from the lines and pumps.

NOTE: Please purge the solvent supply tubings of piston pump and syringe pump if they have not been used in a while.

The purging of the system is done with the syringe pump. To completely purge the system, the following 3 purge procedures have to be performed (Figure 60).

1) Solvent (section 5.7.1, page 51)
2) Sample loop (section 5.7.2, page 54)
3) Syringe pump (section 5.7.3, page 57)

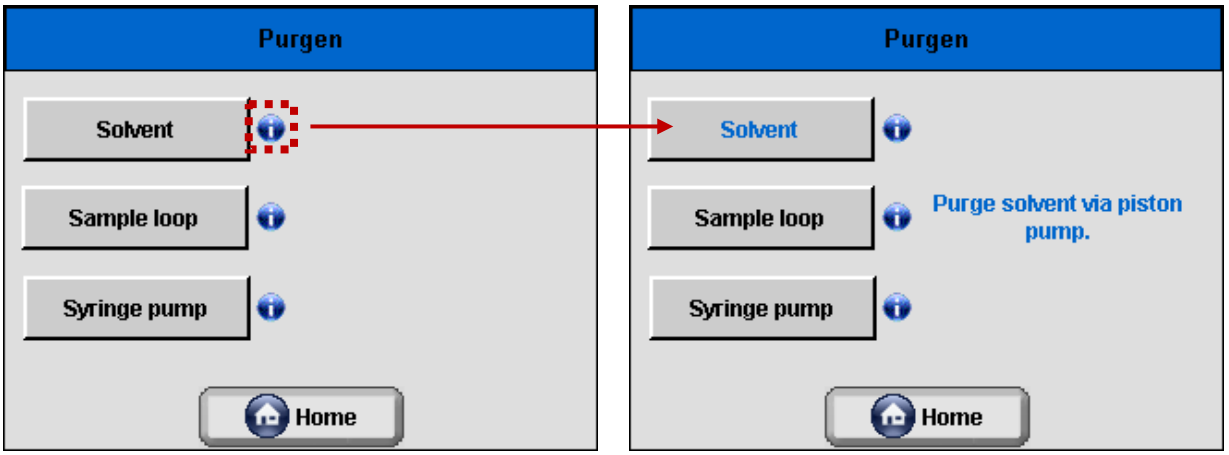


Figure 60: Three different types of purging. If you press the Info button (left screenshot), a note (right screenshot) will appear explaining the respective venting procedure.

5.7.1 Solvent

When venting "solvents" (Figure 61), the piston pump and the tubes fitted in the solvent valve fills are purged at the same time.

Figure 61: Purge solvent via pump.

Generally, a solvent *("n-Hexane"*, *"Toluene"*, *"DCM/n-Hexane (50:50)", "DCM/n-Hexane (20:80)"* or *"All"*) needs to be selected for purging (corresponding button is illuminated according to the colour coding used for the tubes, Figure 62 right), otherwise the process cannot be started (the *"Start"* button is not visible, see Figure 62 left and centre).

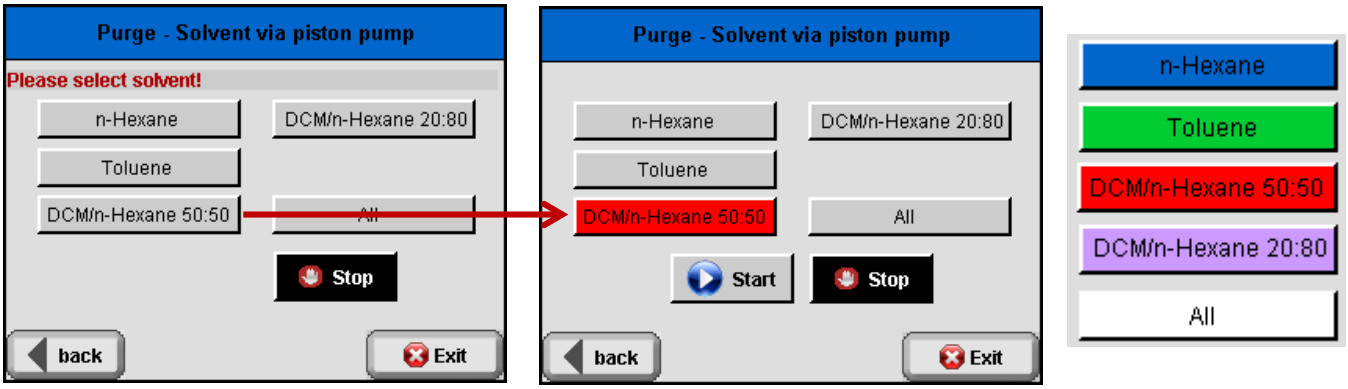


Figure 62: Venting solvent lines via piston pump. After solvent selection (left), the "Start" button (centered) will appear. Right: colour coding of tubings.

The button "*All*" defines the process of filling the solvent lines in the order of: 1. Toluene, 2. DCM/n-hexane (50:50), 3. DCM/n-Hexane (20:80) and 4. n-Hexane.

INFORMATION: If more than the four standard solvents are used (see Settings), the corresponding lines need to be purged, too.

After the process is started (press of the "*Start*" button), the following announcement appears on the display.

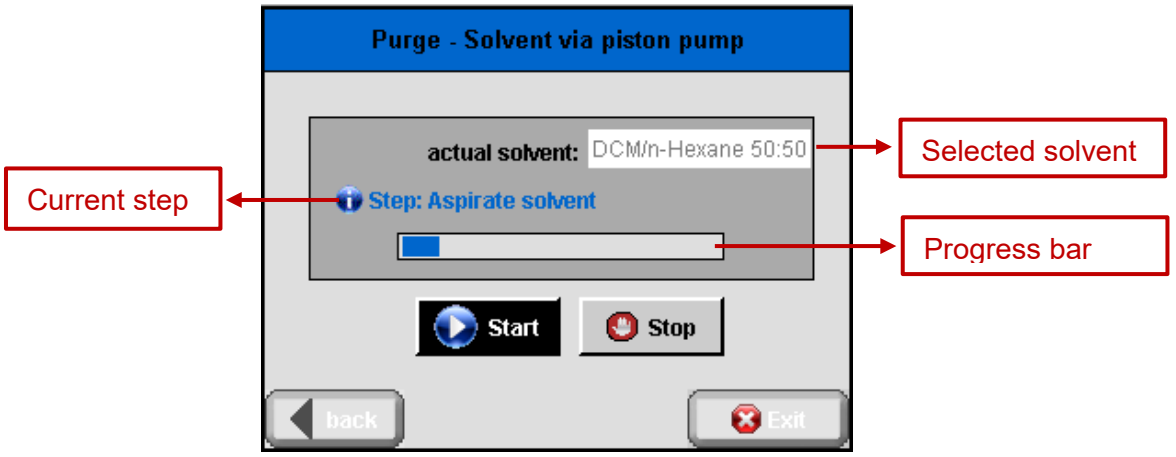


Figure 63: Display shown during solvent purging.

Purging (comply one solvent) involves the following steps:

1) Aspirate solvent from the solvent bottle, via: solvent valve → piston pump → distribution valve → syringe pump.
2) Empty syringe pump → waste

By pressing the *"Stop"* button, the purge process can be interrupted (see Figure 64).

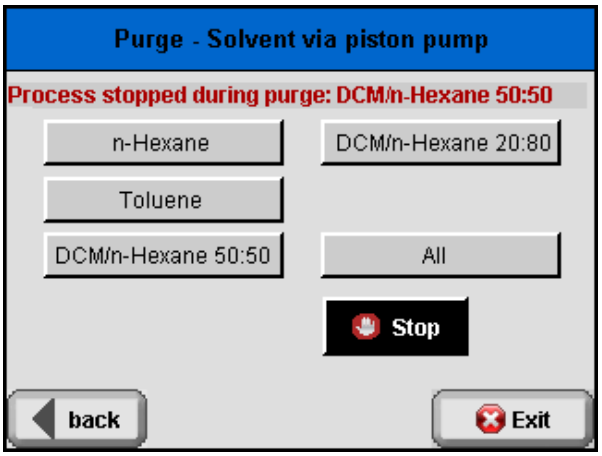


Figure 64: Window if the purge process is interrupted.

After stopping, the purge process can be continued with the following options:

I) Continue process
II) Start another process by choosing a different solvent.
III) Exit

Note on item I) Continue process

To continue the same process, choose the same solvent again and press the *"Start"* button. The system will continue with the processing at the point of interruption.

Note on item II) Start another process

Select a different solvent and press the *"Start"* button. The interrupted process will be discontinued and a new process with the selected solvent will be started. If some solvent had remained in the syringe pump, the new purging step will start with emptying the syringe pump. During this time *"Start"* and *"Stop"* button will appear in black and are inactivated (Figure 65).

Figure 65: Display with inactive buttons.

INFORMATION: While the syringe is emptying, all buttons are inactive.

Note an item III) Exit

You can only exit the purge window if the solvent of the interrupted process was n-Hexane. If the process was interrupted while purging with another solvent, the system needs to be reconditioned first. This is done automatically on leaving the purging window (see Figure 66).

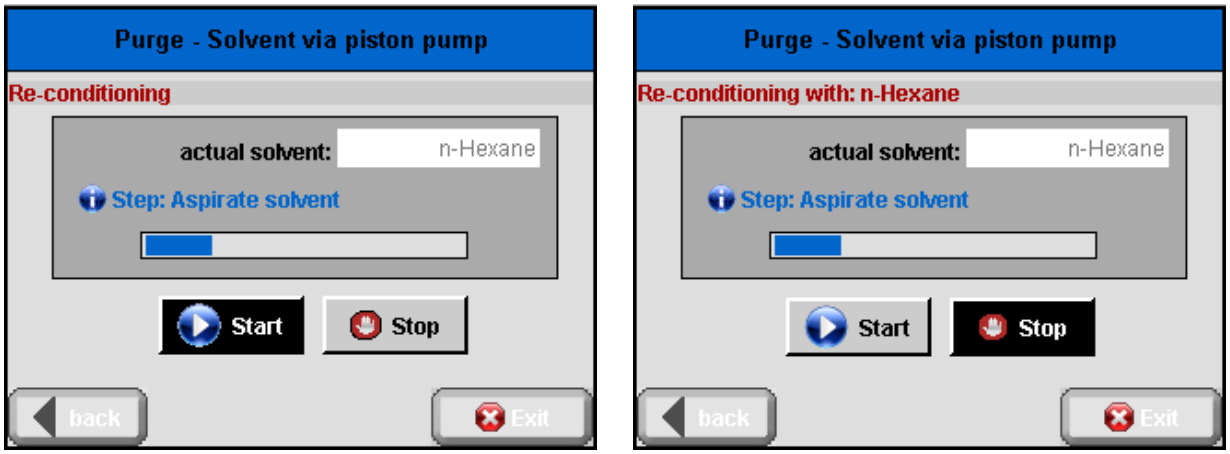


Figure 66: Reconditioning.

After the purging process, the solvent line should be completely filled with solvent. If there is still air in the line, purging needs to be repeated.

INFORMATION: You can only leave the purge window, if the last purging solvent was n-Hexane or if the system was reconditioned with n-Hexane. This prevents any residual Toluene, DCM/n-hexane (50:50), or DCM/n-hexane (20:80) in the lines or syringe pump.

A list of possible errors during the purging process can be found on page 58.

5.7.2 Sample Loop

The following purge process is used to fill up the sample loop with solvent and to remove all air from the sample loop (Figure 67). Before the sample loop can be purged, the lines to the syringe pump have to be purged first (see section 5.7.3).

Figure 67: System components to purge sample loop.

For sample loop purging, the solvent n-Hexane is pre-selected and cannot be changed (compare Figure 68). The volume of 22 mL is also pre- selected to ensure that the purging of the sample loop is fully completed (actual sample loop volume is 20 mL).

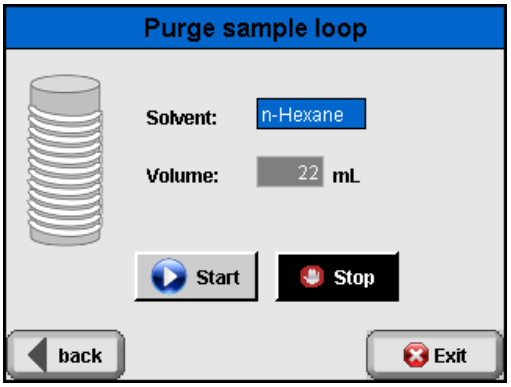


Figure 68: Purging of the sample loop.

INFORMATION: The purging volume of the sample loop can only be changed in the internal section of the software.

To start the process, press the *"Start"* button.

1) The solvent is sucked directly into the syringe pump.
2) The solvent flows through the sample loop into the waste.

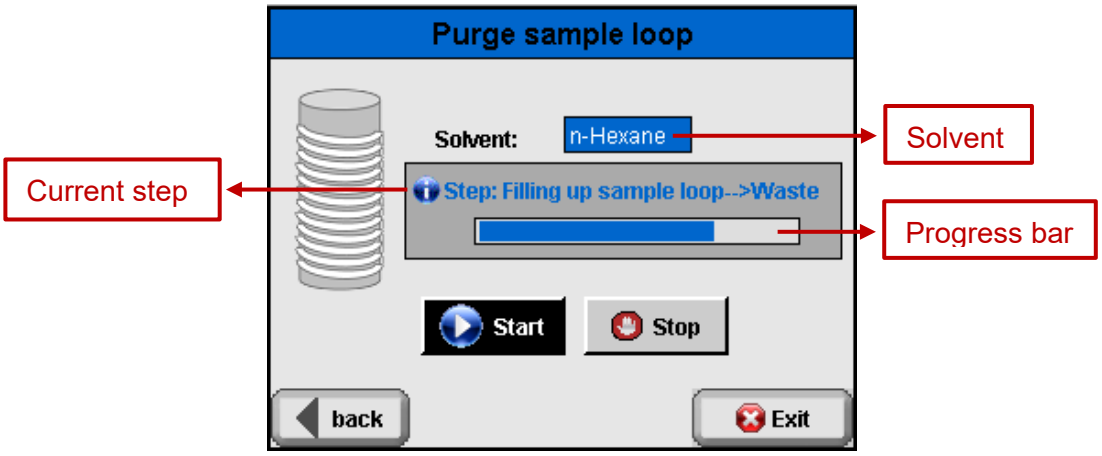


Figure 69: Process fill sample loop.

The process can be stopped by pressing the *"Stop"* button (Figure 70).

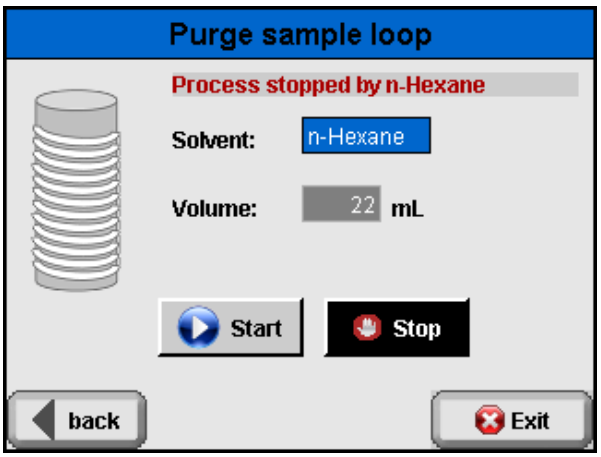


Figure 70: Interruption process.

Two options exist to proceed:

I) Continue process
Press the *"Stop"* button again and the process will continue with the process step at which it had been interrupted.

II) End process
The process is stopped completely, if the purge window is left by pressing the *"back"* button (Figure 70).

The process is successfully completed once no air is left in the sample loop. If air bubbles are still present, the process has to be repeated.

A list of possible errors during the purging process can be found on page 58

5.7.3 Syringe Pump

During the purging of the syringe pump, the 3 colour-marked solvent lines are vented (Figure 71).

Figure 71: Syringe pump.

To start the process, select the solvent and press the *"Start"* button.

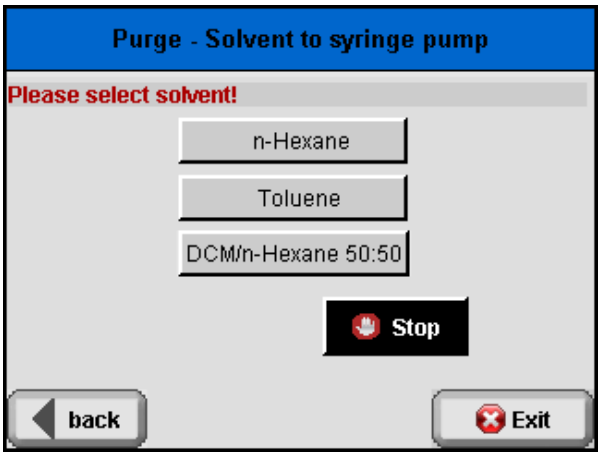


Figure 72: Purging syringe pump.

The purging for each solvent consists of the following steps:

1) The solvent is drawn into the syringe pump.
2) The syringe pump ejects the solvent into the waste.

After the purging process, the solvent line should be completely filled with solvent. If air remains in the line, the process needs to be repeated.

INFORMATION: You can only leave the purge window if the last purging solvent was n-Hexane or if the system was reconditioned with n-Hexane. This prevents any residual Toluene or DCM/n-Hexane (50:50) in the lines or syringe pump.

If an error occurs during venting, the system will be immediately stopped and put into pause mode. The triggering fault is then displayed in a separate window.

Possible error messages during venting:

- Overpressure (page 121)
- Overpressure syringe pump (page 122)
- Waste full (page 123)
- Error valve (page 125)
- Leakage (page 126)
- Lock column tower (page 127)
- Close door (page 128)
- Error syringe pump (page 129)
- Error Init SP (page 130)
- Error Initialization SP (page 131)
- Error SP valve overload (page 135)
- Error SP plunger move (page 136)

5.8. Rinse

Every sample process may be followed by a short rinsing (Figure 74) whereby different system components can be rinsed. To reach the rinse window, select the *"Rinse"* button in the main window. In the "RINSE"- window, 3 different options are available (Figure 73) and additional information is shown depending on which components have already been rinsed (Figure 74).

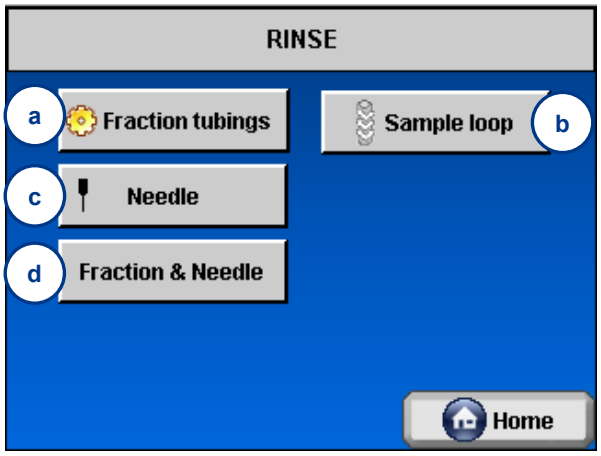


Figure 73: Rinse-window if components have already been rinsed.

a) Fraction tubings
(Section 5.8.1, page 60)

b) Sample loop
(Section 5.8.2, page 62)

c) Needle
(Section 5.8.3, page 63)

d) Fraction & Needle
(Section 5.8.4, page 66)

Below, this rinse-window is shown after a sample run. Steps marked in red highlight the components that need to be rinsed.

The difference between "*Home"* and "*No Flushing"* is that "*Home"* skips the rinsing process and the needle rinsing until the process button is clicked the next time and the rinse window will appear in due course. "*No Flushing*" on the other hand causes that no further prompts for neither a rinsing process nor needle rinsing will appear.

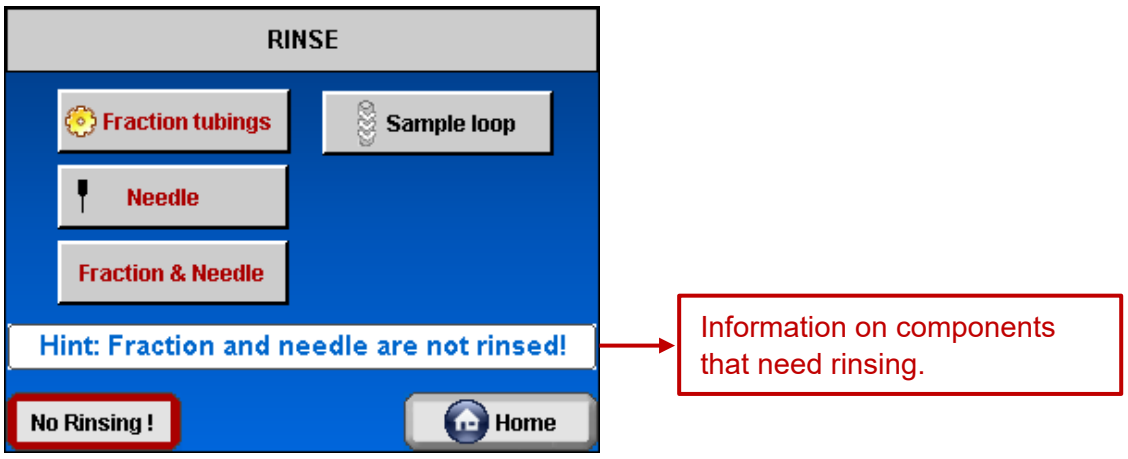


Figure 74: Rinse window naming components (marked in red), which need to be rinsed.

INFORMATION: If the sample process was interrupted during the sample transfer an additional button [Back transfer] is shown in the rinse window. For more information see section Backtransfer of sample .

5.8.1 Fraction tubing

The system rinses column 1 and the two fraction tubings with the solvent n-Hexane and dries the columns.

Preparations for the rinsing of the system:

For rinsing, the columns need to be replaced with dummy columns (shown in Figure 75).

Dummy column:
- Position 1: Universal column
- Position 2: Alumina column
- Position 3: Carbon column

Figure 76: Dummy columns and their respective position in the column tower.

Note for inserting the dummy columns:

➔ Insert each dummy column only in its intended position (see Figure 76).

➔ To prevent an impact, columns 1 and 2 must be inserted into the column tower with the hole visible at the front (Figure 76).

Figure 75: Correctly inserted dummy columns in the column tower.

ATTENTION: When replacing columns with dummy columns, solvent may leak out in an uncontrolled manner. Please use hand and eye protection.

Safety note: The opening and closing of the column tower may only be undertaken with the front door closed. If the door is not closed, a window will appear with the request to close the door before you open/close the column tower.

Figure 77: Prompt to close the door.

After insertion of the dummy columns, all fraction flasks (on the front side of the device) need to be connected to a waste container.

The rinsing process is started with "*Start*" (Figure 78).

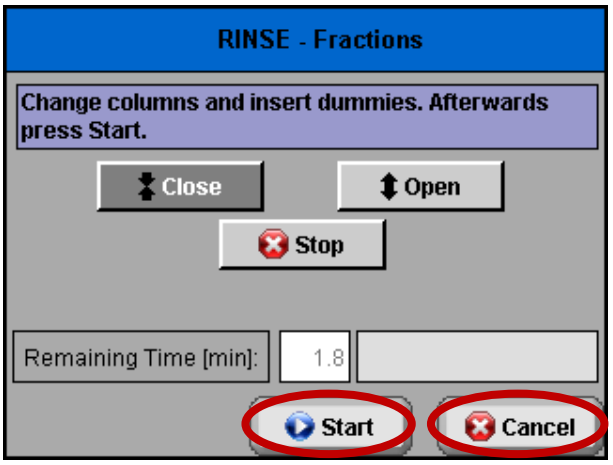


Figure 78: Rinsing window with the option to start a rinsing process with "Start" or to leave the rinsing window without rinsing with "Cancel".

NOTE: A new method can be set up for an individual rinsing process, i.e. rinsing the system only (see Chapter 5.4 Menu Method).

Safety advice:
It is not possible to open the column tower during rinsing (marked by the locked "*Open*" button), shown in Figure 79.

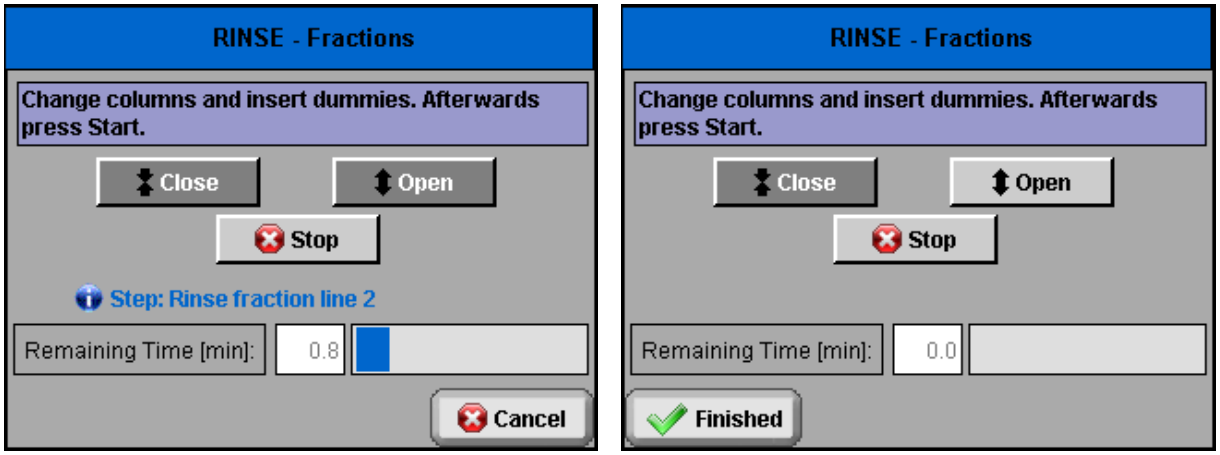


Figure 79: Left screenshot: Locked "Open" button during rinsing. Right screenshot: Finished rinsing process with the option to open the column tower.

If an error occurs during the rinsing process, the system will be stopped immediately and put into pause mode. The triggering fault is then displayed in the rinsing window.

Possible error messages during rinsing:

- Overpressure (page 121)
- Waste full (page 123)
- Error valve (page 125)
- Leakage (page 126)
- Error pump (page 132)

5.8.2 Sample Loop

The following shows the window for rinsing the sample loop with a choice of different solvents (Figure 80).

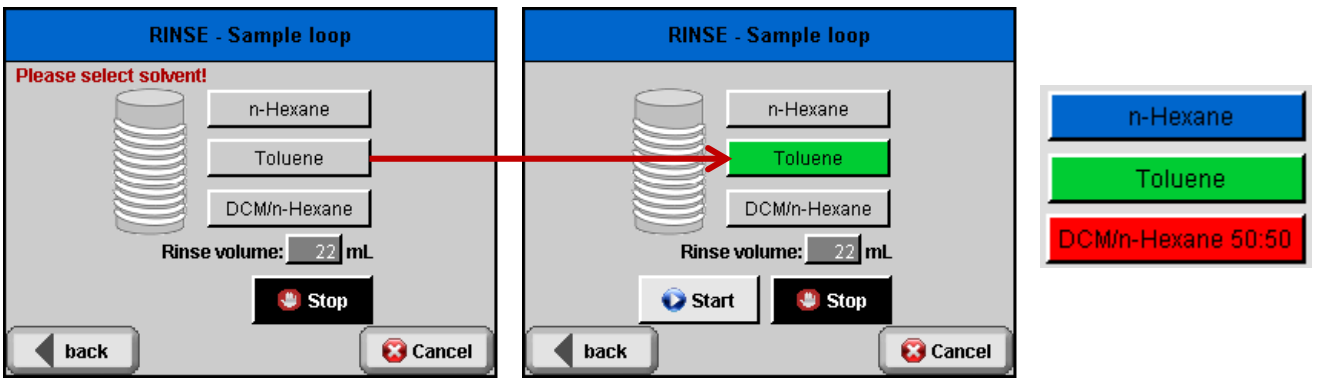


Figure 80: Rinsing of the sample loop. After choosing a solvent (left), the "Start" button will appear (middle). The solvent colour code is show on the right.

All following steps in the software are similar to those in the section for purging solvents (see section 5.7.1).

These are:

1) The solvent is drawn into the syringe pump.
2) The solvent is pumped through the sample loop into the waste.

Afterwards, the sample loop should be rinsed and reconditioned with n-Hexane.

INFORMATION: You can only leave the purge window if the last purging solvent was n-Hexane or if the sample loop was reconditioned with n-Hexane. This prevents any residual Toluene, DCM/n-Hexane (50:50) or DCM/n-Hexane (20:80) in the lines or syringe pump. For how to recondition the sample loop see Section 5.7.1.

If an error occurs during the process, the system will be stopped immediately and put into pause mode. The triggering fault is then displayed in the rinsing window.

Possible error messages during rinsing of the sample loop:

- Overpressure (page 121)
- Overpressure syringe pump (page 122)
- Waste full (page 123)
- Error valve (page 125)
- Leakage (page 126)
- Error syringe pump (page 129)
- Error init SP (page 130)
- Error initialization SP (page 131)
- Error SP valve overload (page 135)
- Error SP plunger move (page 136)

5.8.3 Needle

In order to rinse the needle from the inside and the outside, an empty vial has to be placed into the sample holder. Additionally, the sample holder has to be placed below needle (see Figure 81).

Figure 81: Rinse position of needle.

In order to start the preprogramed rinse process, press the "*Start*" button. A change of the needle rinse process can only be made from the internal section of the software (rinse parameter Chapter 10.3.1).

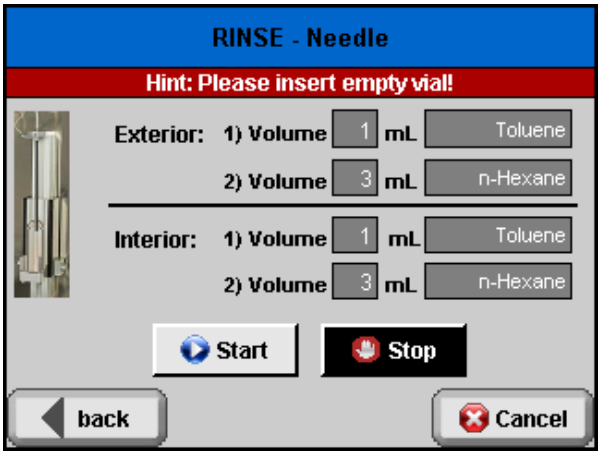


Figure 82: Default rinse program of needle.

After pressing the "*Start*" button the following display will appear.

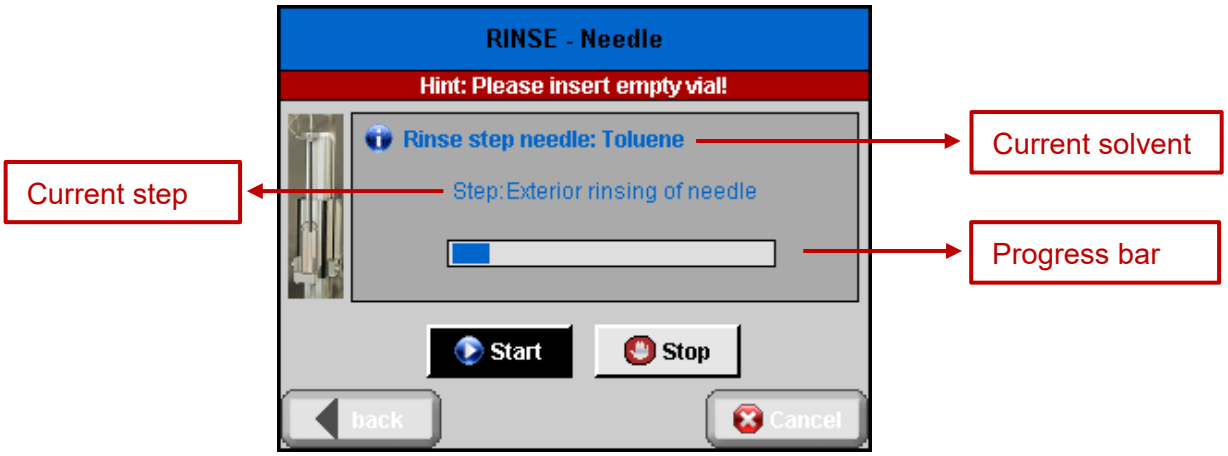


Figure 83: Process announcement for the needle rinse.

The following steps are part of the process:

1) The first solvent is drawn into the syringe pump.
2) Outside rinsing of the needle via the small capillary holes in the needle.
3) Inside rinsing of the needle.
4) The second solvent (n-Hexane) is drawn into the syringe pump.
5) Outside rinsing of the needle via the small capillary holes in the needle.
6) Inside rinsing of the needle.
7) Aspirate air buffer.

If necessary, the process can be interrupted by pressing the "*Stop*" button (see Figure 84).

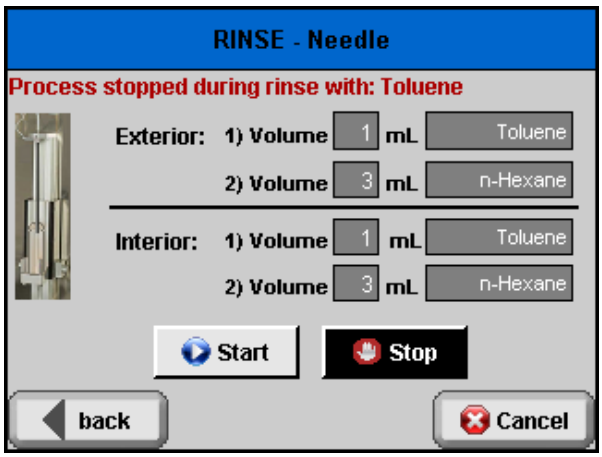


Figure 84: Interrupted needle rinse process with the information of solvent used at the time of interruption.

Afterwards, the process may be continued with either of the 2 following options:

1) Continue process:
   Press the "*Start*" button again, and the process will continue with the process step at which it had been interrupted.

2) Cancel process:
   You can abort the process by leaving the window with either the "*back*" or "*Cancel*" button. If the process was aborted during the cleaning with Toluene, the system will automatically recondition itself with n-Hexane (see Figure 79 and Figure 80).

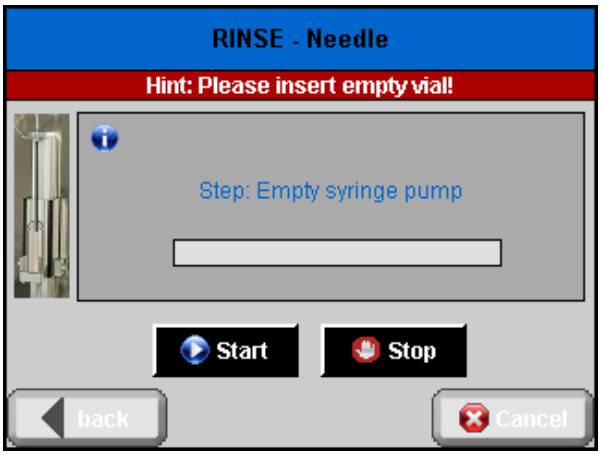


Figure 85: Emptying of the syringe pump.

INFORMATION: As long as the syringe pump is emptying, the buttons are black and inactive.

You can only leave this window once the reconditioning process is complete.

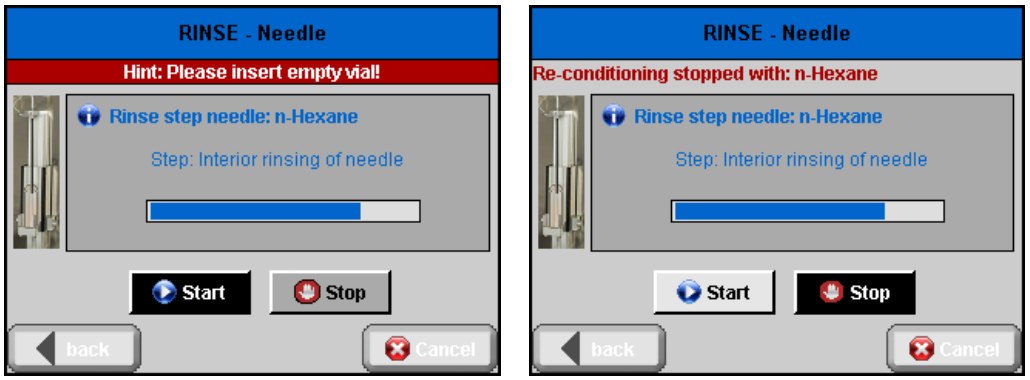


Figure 86: Reconditioning after the needle rinse was interrupted.

If an error occurs during the needle rinsing process, the system will be stopped immediately and put into pause mode. The triggering fault is then displayed in the rinsing window.

Possible error messages during needle rinsing:

- Overpressure (page 121)
- Overpressure syringe pump (page 122)
- Waste full (page 123)
- Error valve (page 125)
- Leakage (page 126)
- Error syringe pump (page 129)
- Error init SP (page 130)
- Error initialization SP (page 131)
- Error SP valve overload (page 135)
- Error SP plunger move (page 136)

5.8.4 Fraction & Needle

Include a combined process for simultaneous rinsing of fraction tubing and needle (Figure 87).

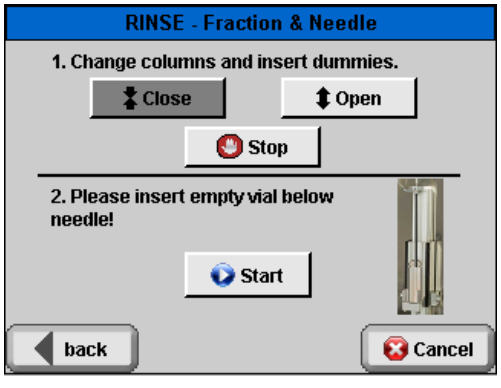


Figure 87: Process window for combined rinsing.

Preparations for rinsing the system:

1) For rinsing, the columns have to be replaced with dummy columns and all fraction flasks (on the front side of the device) need to be connected to a waste container.
   - For more information about using dummy columns see chapter 5.8.1 Fraction tubing .
2) Place an empty vial into the sample holder and place the holder below the needle.

The rinsing process is started with *„Start“* (Figure 87).

The system components thus are rinsed (Figure 88), analogous to the separate rinsing of the fraction tubing and needle. For more detailed information, please refer to chapter, 5.8.1 Fraction tubing and 5.8.3 Needle .

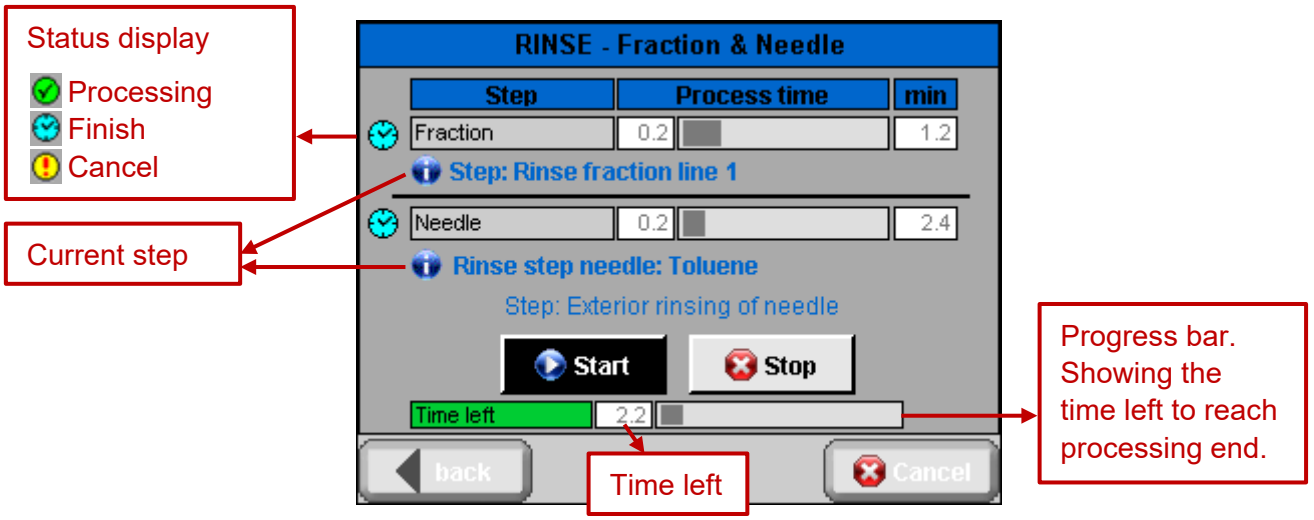


Figure 88: Activated rinsing process.

The simultaneous process can be interrupted at all times by pressing *„Stop“*. You can only leave the window by “*Cancel*” after the needle has been reconditioned with n-Hexane (see Figure 89, left). The status display (described in Figure 88) informs about the current status.

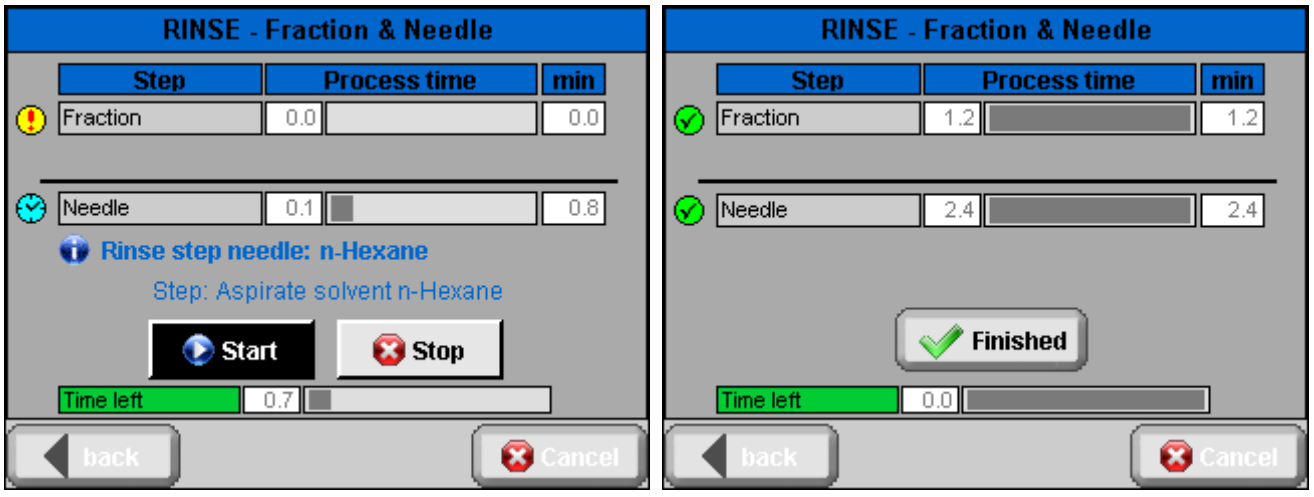


Figure 89: Left: Canceled rinsing process. The rinsing of the fraction tubing is terminated and reconditioning of needle started. Right: The complete simultaneous rinse process has been finished.

5.9. Combine

The „Combine“ button (see Figure 3) combines the purging of the pumps with the rinse steps of the needle, sample loop and fraction lines. After pushing the button, the following steps will be performed automatically:

1. Purging of all solvent lines going to the two pumps
2. Rinsing of the needle and fraction line
3. Rinsing of the sample loop

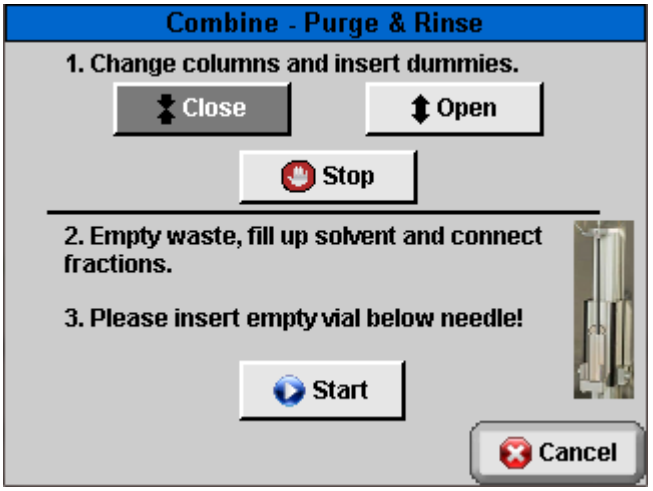


Figure 90: Combi-Process of purging the solvent lines and rinsing of the needle, fraction lines and sample loop.

To start the Combi- Process the following steps have to be done:

1. Place the Dummy columns into the columns tower
2. Place the fraction lines into a waste container
3. Make sure the waste bottles are empty
4. Place an empty vial below the needle

Press the “Start button” to start the process. (Figure 91).

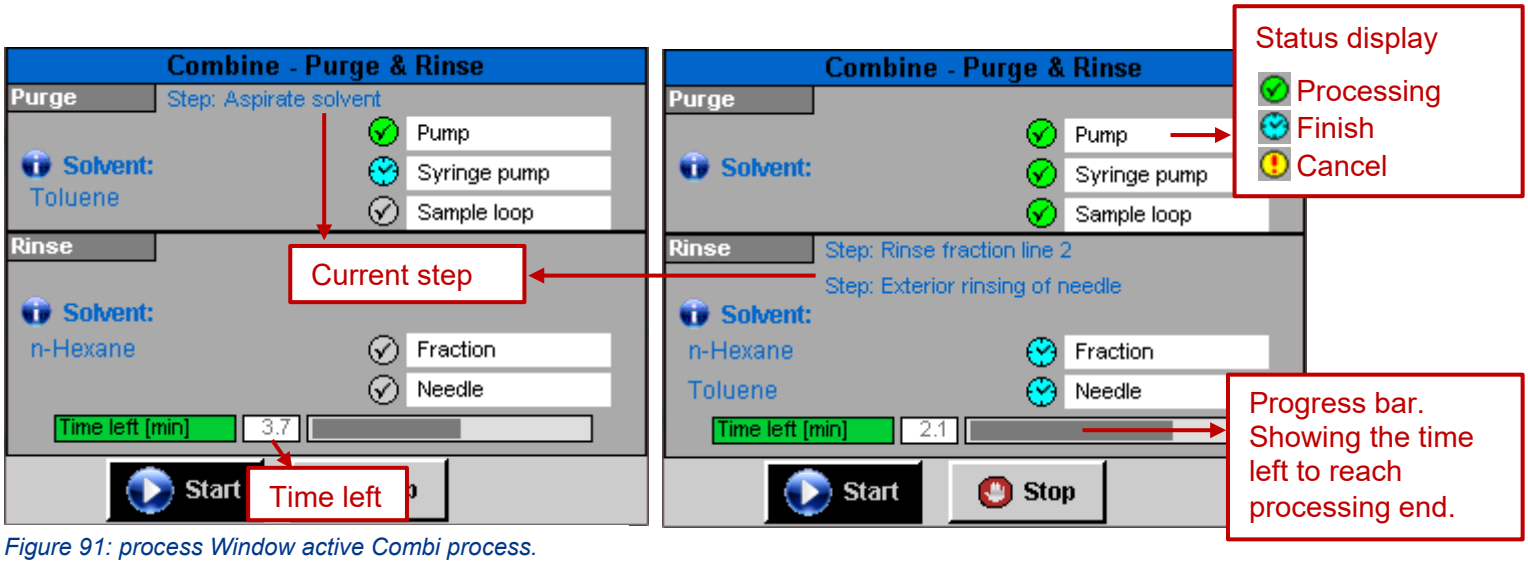


Figure 91: process Window active Combi process.

The process starts (Figure 91, left) with purging of all solvent lines of the syringe and piston pump. (See chapter 5.7.1 Solvent und 5.7.3 Syringe Pump). Afterwards the sample loop is rinsed with n-hexane. (chapter 5.7.2 Sample Loop). At the end of the process also the sample needle and the fraction lines are rinsed. (see chapter, 5.8.1 Fraction tubing und 5.8.3 Needle)

The process can be stopped (“Stop-button”) at every step in the process. Nevertheless the procees will be stopped only after the pump, syringe pump and needle are reconditioned with n-hexane
The status indicator shows the position in the process (described in in Figure 91)

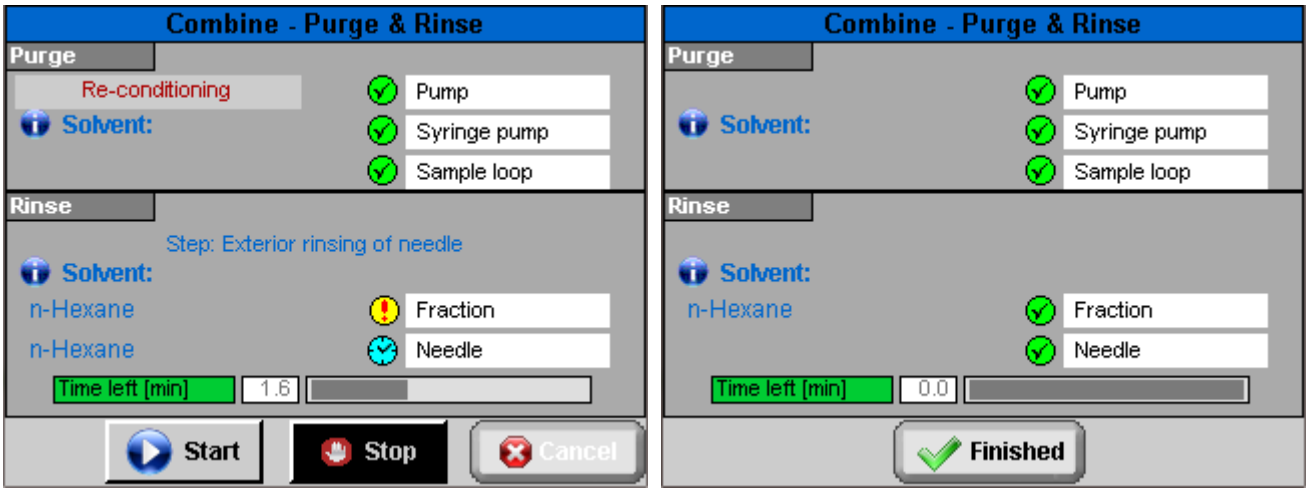


Figure 92: Left. A interrupted process is shown. Right a fully processed “Combi run” is shown.

6. Heating Control Unit

To control the three heating zones an additional control unit is placed on top of the instrument (as shown in Figure 93).

There are three panels, one for each heating zone (as shown in Figure 94):

- **TUBE-HEATING**
- (Tube from the needle to the sample loop and from the sample loop to the first column)
- **SAMPLE-LOOP**
- **VIAL**

To turn ON/OFF the control unit use the switch on the right hand side.

Figure 94: Heating control unit.

The connections are clearly marked with labes on the cables and plugs.The power supply has a voltage of 25V/DC (see Figure 95).

Figure 95: Wiring of the heating control unit.

6.1. Adjusting the Temperature

Turn on the control unit (I) and wait a few seconds until the display shows the current temperature of each heating part (as shown in Figure 96). The instrument will automatically heat the parts up to the previous set temperature.

Figure 96: Indication of the current temperatures.

After pressing the button “P” (programm) on the panel, the display will show the inscription “SP” (Set Parameter) and the set value (Figure 97).

Figure 97: Swapping indications after pressing the “P”-button.

By pressing the arrow keys (UP/DOWN) the user is able to adjust the set temperature in 0.1°C steps. The set temperature will be automatically saved after a few seconds or by pressing the “P”-button again in order to confirm.

NOTE: Please be aware that the heating control unit is not able to cool the system down, its purpose is generating and controlling heat only.

7. Report

The activated report function creates reports for samples that have been prepared by using the DEXTech Heat. The reports generated aid quality assurance in an analytical laboratory. For reporting, the data must be transferred from the device to a PC using for example a USB stick (the USB stick supplied may be used for this purpose; see section Chapter 7.1). After data transfer from a USB stick to the PC, report generation can be commenced on the PC (see Chapter 7.2).

7.1. Usage of the DEXTech Heat Software

The "*Report*" button is located at the main menu (see Figure 98). After the first sample preparation, the "*Report*" button becomes active and the function can be executed.

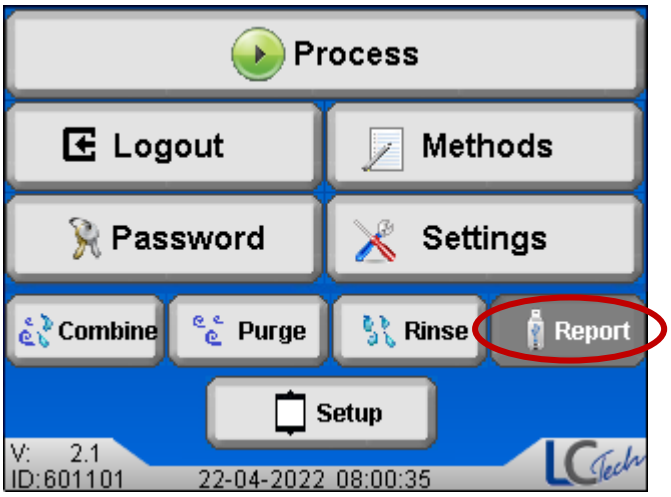


Figure 98: "Report" button.

To start a sample, press the "*Process*" button and wait until it has been successfully completed. Then press the *"OK"* button (see Figure 99, left screenshot) and the associated sample data will be recorded and saved. If a report is required, return to the main page and press the "*Report*" button (see Figure 99, right screenshot).

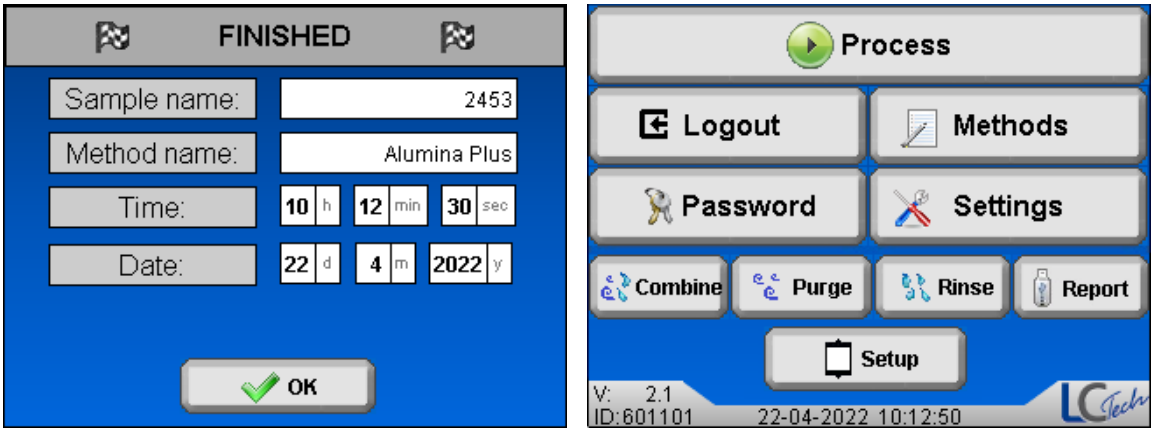


Figure 99: Enabling the option for data transfer. After the completed sample preparation (left), the "Report" button on the main page (right) will appear in the same shade of grey as the other buttons

7.1.1 Preconditions for the DEXTech Heat Data Transfer

Successful data transfer onto a USB stick is only warranted for the use of a standard USB 2.0 stick. Only the USB stick supplied may be used for this purpose. To upload data, insert the USB stick in the USB port on the right-hand side of the unit (Figure 100).

Figure 100: USB port on the DEXTech Heat.

7.1.2 Data Transfer to a USB Stick

Press the "*Report*" button on the main page. Following this, an error message will be displayed on the screen. This indicates that no USB stick was found on the device (see Figure 101).

Figure 101: Error message indicating that no USB stick has been found on the device.

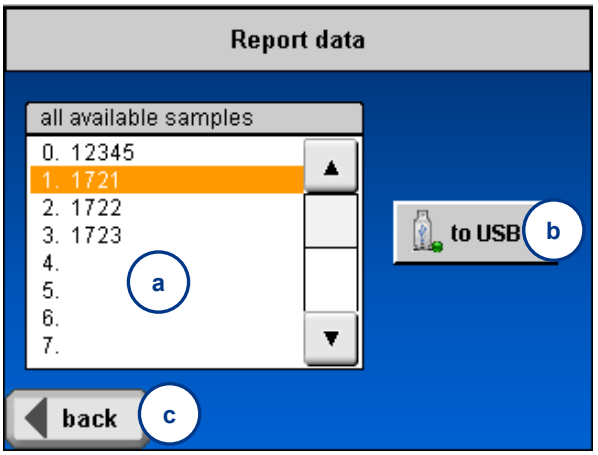
Now, a USB stick must be inserted into the device USB port (see Figure 100). Once the USB stick is connected, a report page (see Figure 102) will appear. From here, data transfer (of all previously stored sample data) can be initiated by clicking the button "*to USB*". All sample data stored and subsequently transferred are contained in the list.

Important: If a USB stick is connected to the DEXTech Heat and data transfer is activated in the software (button pressed), the USB stick may only be removed from the DEXTech Heat when prompted (see Figure 99, on the right).

List of all existing sample preparations.

a) Button for activation of data transfer of all listed sample data onto a USB stick.

b) „*back*“ button to return to the main page.



After pressing "*to USB*", a holding page will be appear showing the progress of the data transfer (see Figure 103, left screenshot). A screen message will confirm the successful completion of the data transfer (Figure 103, right screenshot). Thereafter, the USB stick may be removed from the device.

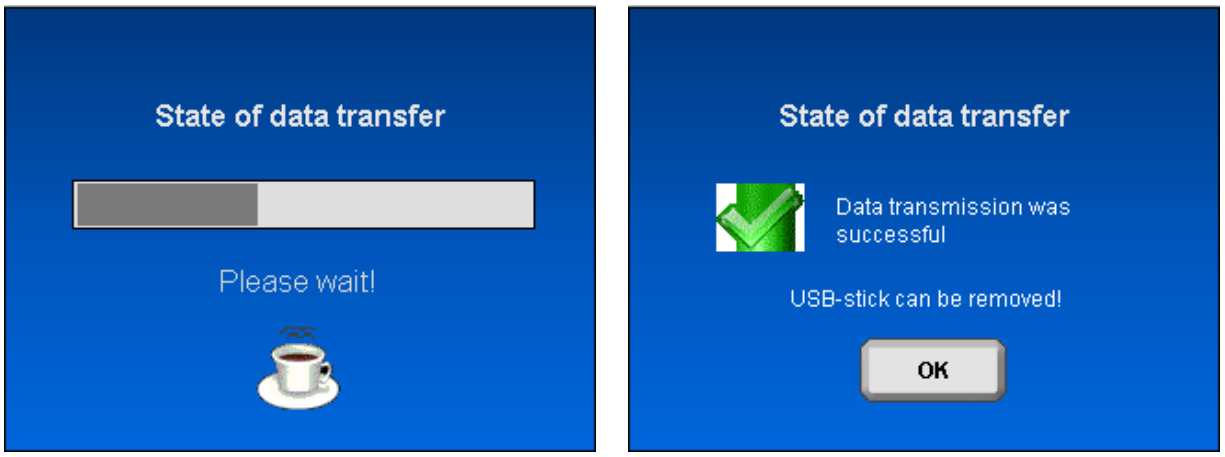


Figure 103: Progress of data transfer. Progress screen is show on the left, and completion of data transfer on the right.

On return to the main page, the colour of the “*Report*” button will be light grey again. This indicates that there are no data remaining to generate a report for, as the transfer has already taken place.

On the USB stick, you will now find a folder called "DEXTechHeat" followed by the six digit serial number of the device (*ID* display on the main page of the device). In this automatically created folder is a file named "*Report.dio*", which contains all samples data from the executed sample preparations.

INFORMATION: All future data transfers from this DEXTech Heat device to the USB stick will be stored in the same folder (with the associated ID). The file name will be made up of the word “Report” plus a sequential number (for example: Report1.dio). Should the same USB stick be used for several different devices, a new folder called “DEXTechHeat” followed by the corresponding 6-digit serial number” will be created on the USB stick for each of the devices.

7.1.3 Monitoring the Report Data Memory

The report data storage space is limited to 50 sample preparations. One storage space is required per sample preparation (for exceptions see information note below). As soon as only three free spaces are left in the memory (see Figure 104) a notification will be displayed indicating the number of samples that can still be processed without overwriting the existing sample data.

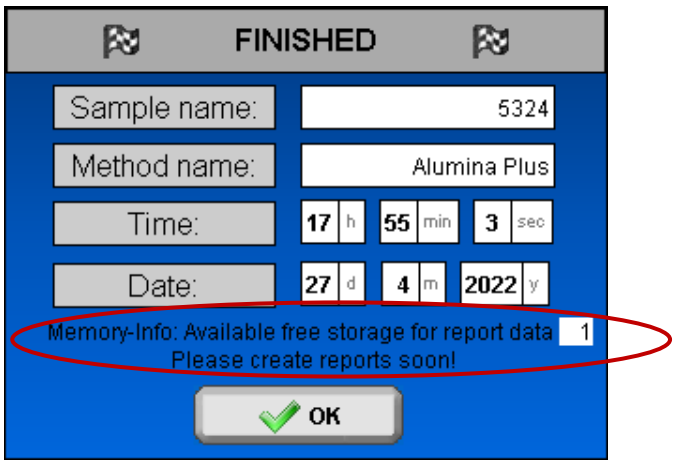


Figure 104: Display of available storage after finish of the current sample preparation (here: one).

INFORMATION: If one method is applied for conditioning and another separately for fractionation within one sample preparation, then the sample data will be saved in two separate reports.

All interrupted or cancelled samples will also be stored as a report.

If storage space is full, data transfer should be undertaken in order to prevent overwriting of the already saved report data. However, should a new process get started before data were transferred, a warning as shown in Figure 105 will appear. Confirmation of the warning “Sample data overwrite” with the “*Yes*” button results in overwriting of the oldest set of data. Pressing the “*NO*” button allows for a data transfer to a USB stick before processing further samples.

Figure 105: Warning before saved sample data are overwritten.

INFORMATION: When choosing the sample name, please ensure that this has not been previously used. If a sample name has been used before, a message will appear on screen with the request to choose a different sample name (see Figure 102).

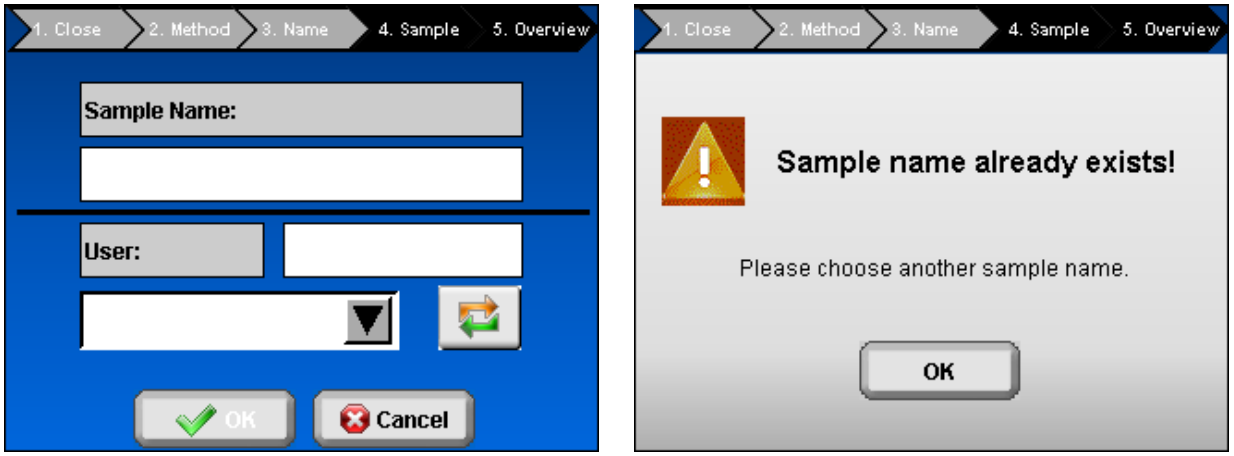


Figure 106: After input of an already existing sample name (left) a message will appear (right) prompting you to choose a different sample name.

7.2. Generating Reports on a Personal Computer (PC)

There is no requirement to install a separate program on the PC in order to generate a report. It is sufficient to copy the entire folder named "*SW_Report*" (located on the supplied USB stick) onto the hard disk of the computer (at any location / path). This folder contains all necessary files that are required by the reporting software.

To generate reports, run the file *"Dioxin.exe"*. This will open a window (see Figure 107), offering the options to either customise *Set-Up* (a) or to generate *Reports* (b).

Figure 107: Main window of the DEXTech Heat software for report generation of sample preparations.

a) Software Settings

In the settings window (shown in Figure 108) the software language can be changed. There is a choice of German (deutsch.lng) or English (english.lng).

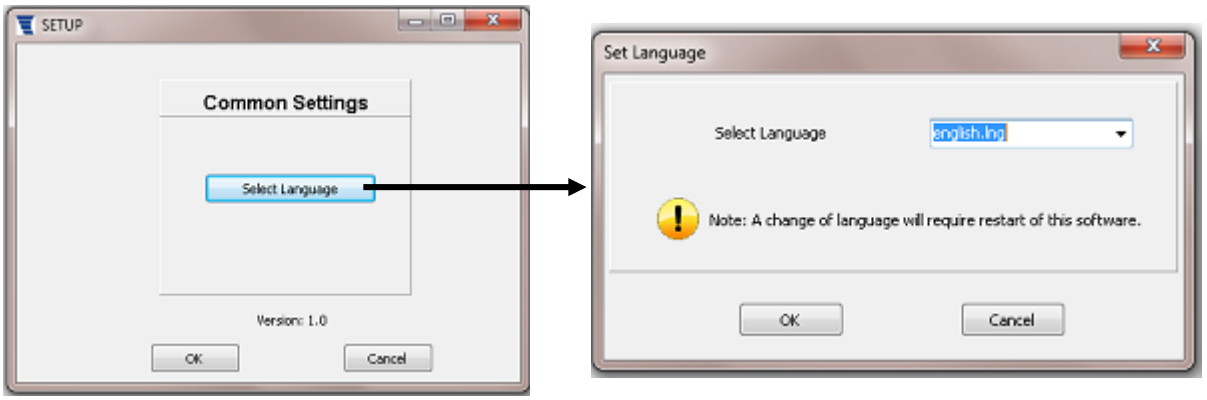


Figure 108: Settings window with tab for language selection: German or English.

If the language is changed, a dialog box will appear (see Figure 109), which indicates the need to restart the software. "*Yes*" will restart immediately; "*NO*" will change the language when the software is started the next time.

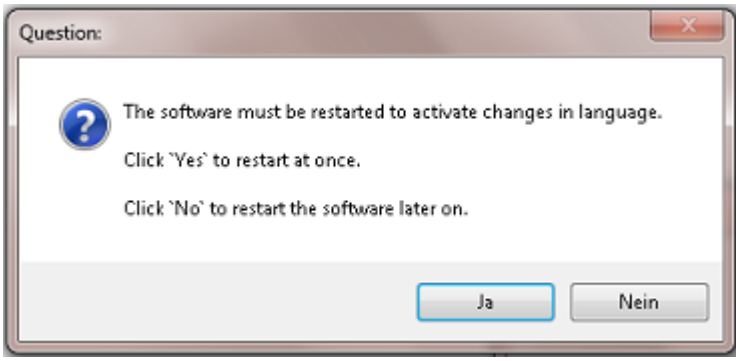


Figure 109: Notification to restart the software after changing the language.

b) Report Generation

Report generation on the PC is only possible, if the DEXTech Heat report data are available on a USB stick that has been connected to the PC (see Figure 110).

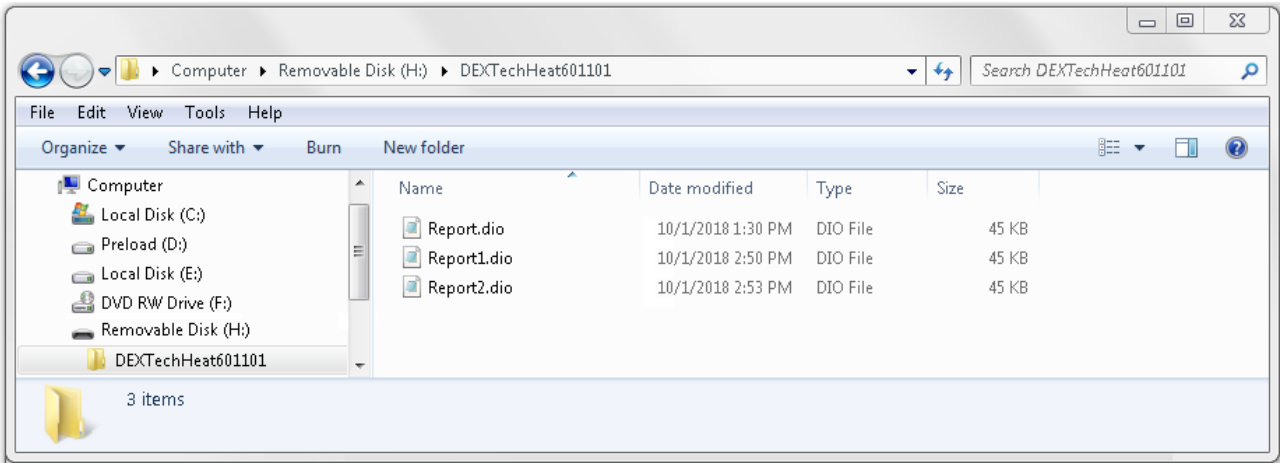


Figure 110: Saved report files (in the folder DEXTechHeat601101) on the USB stick.

Report generation is achieved by clicking the button *"Report"* on the main page (see Figure 107, point b).

INFORMATION: If no USB stick is connected to the PC, the following error message will appear.

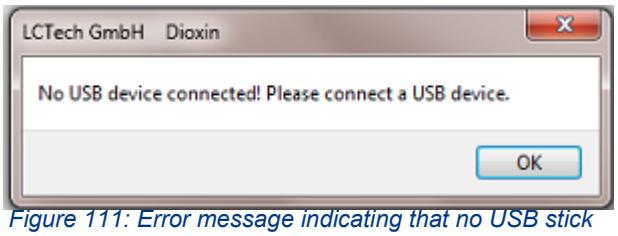


Figure 111: Error message indicating that no USB stick was found on the PC.

The software will automatically detect a connected USB stick. In addition, the software automatically pre-selects the folder containing the sample preparation data required for reporting.

INFORMATION: If there are several DEXTech Heat folders containing sample data, then the folder that is found first (smallest serial number) will be pre-selected.

Important: Once a USB stick is connected and has been selected as path in the software, the USB stick may only be removed from the computer after completion of the report generation.

Then, the report generation page will be displayed (Figure 112).

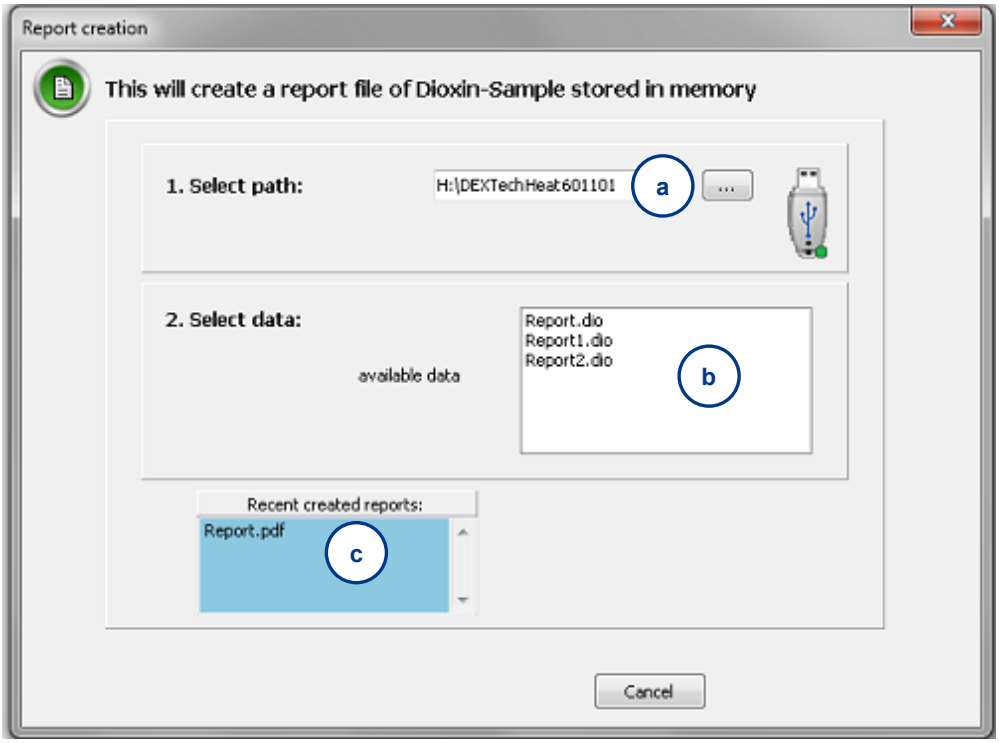


Figure 112: Report generation page with automatically recognised USB stick and folder DEXTechHeat601101 containing the respective r*eport data.*

a) Suggested path for the USB stick and pre-selected folder.

b) List of all files contained in the pre-selected folder with collected sample preparation data (one file corresponds to one set of transferred data from the DEXTech Heat device to the USB stick).

c) List of all previously created reports that have been stored on the computer in the reports file with the 6-digit serial number of the device.

INFORMATION: In the event that no USB stick is found or an incorrect path has been pre-selected, the user has the option to change the path. By clicking the button for path selection (Figure 109, left) a standard Windows dialog box will open (Figure 109, right). Here it is possible to specifically select the path for the connected USB stick and the DEXTechHeat folder with the unique 6-digit number (serial number of the device required).

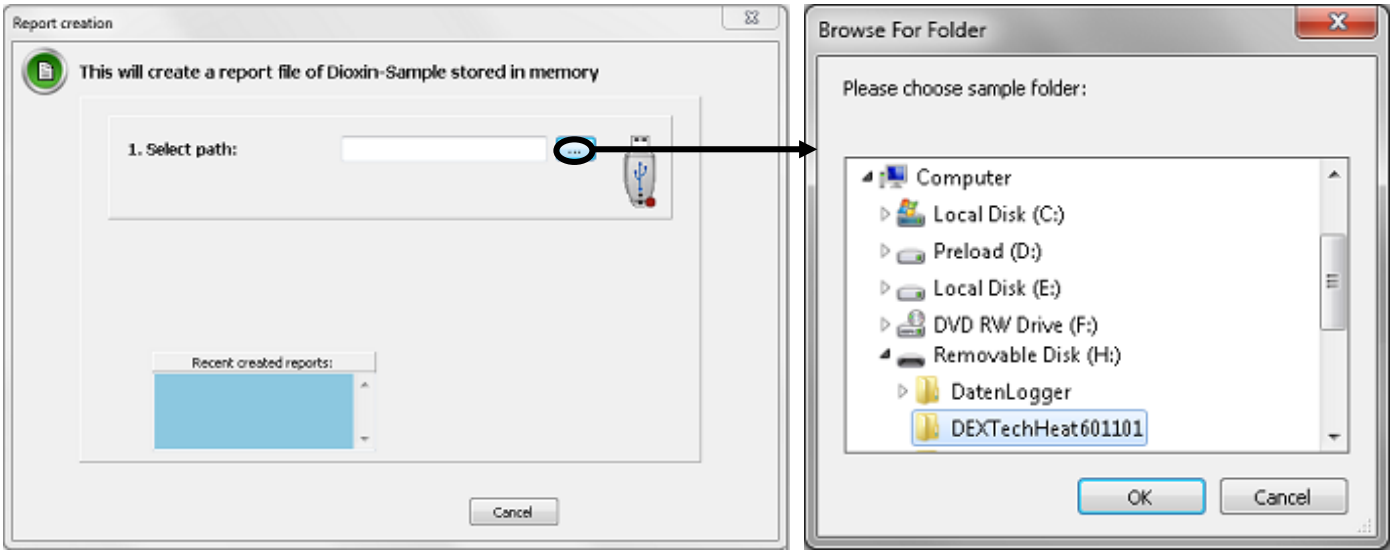


Figure 113: Report generation page (left) and Windows standard dialog box for path selection (right).

After selecting a sample file from the list, a forward arrow will appear which when clicked progresses to the next page (see Figure 115).

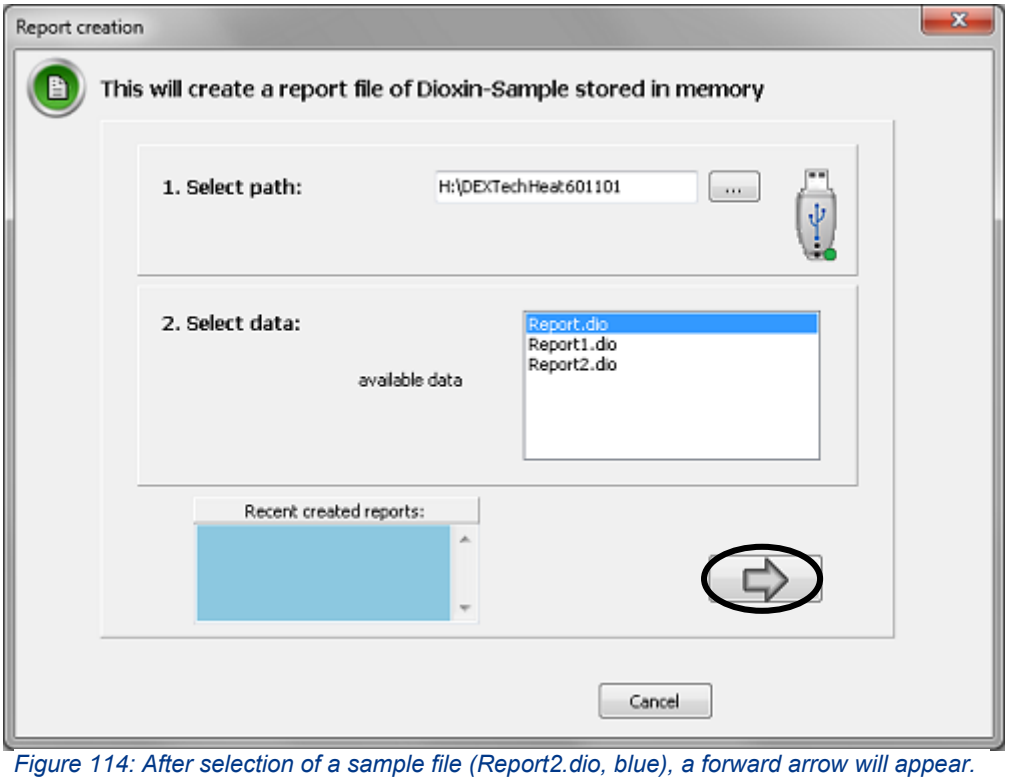


Figure 114: After selection of a sample file (Report2.dio, blue), a forward arrow will appear.

This window (see Figure 114) allows selecting the format in which you wish to save the generated report.

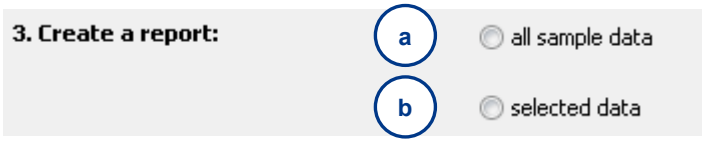


Figure 115: Option for report generation.

a) Save all sample data that are contained in the selected file (e.g. Report.dio) in one PDF file (page 82).

b) Select individual sample data and save in separate PDF files (page 84).

Use the "*Back*" button to return to the previous page.

Option a) collect in one PDF file:

Following selection "a", a forward arrow leading to the next page will appear. There is an option to choose a new name for the file, and if required, to select a different storage path. Moreover, by ticking "show created report", the finished report will be displayed immediately (see Figure 116).

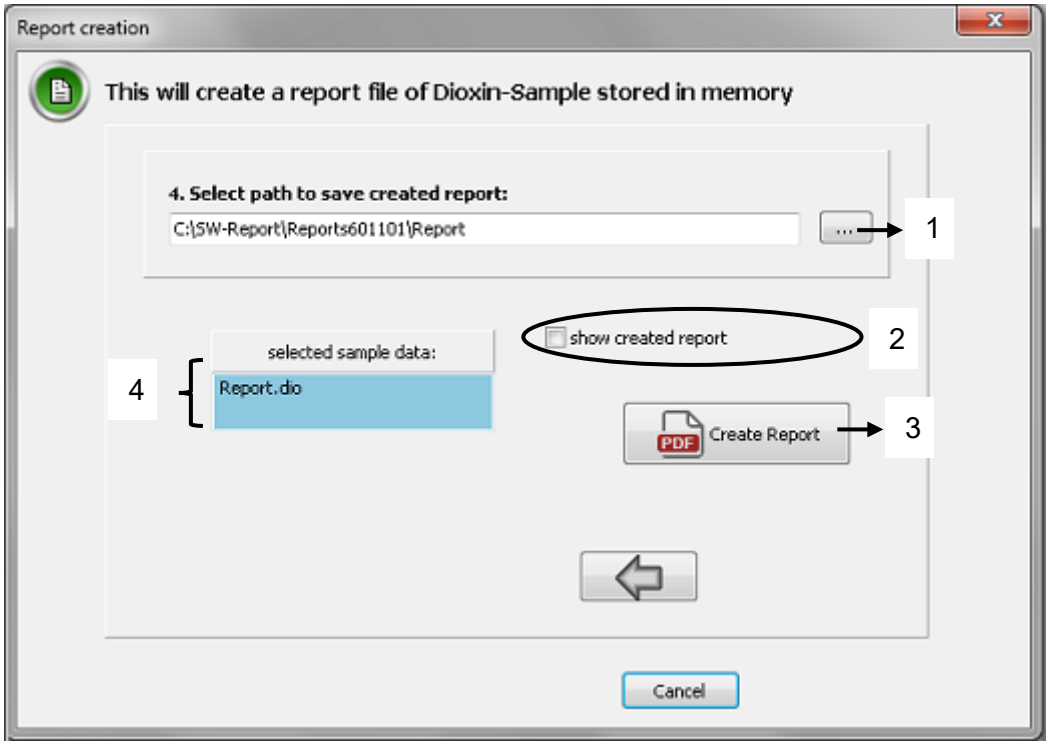


Figure 116: Report generation page with different options.

1) Option to rename the file for the report to be produced, and if required, to change the storage path.

INFORMATION: If the file name was changed, the list (of the most recent reports) will not include this generated report.

2) Option to display the newly generated report immediately.

INFORMATION: The installation of a PDF reader is required for this option.

Should the report not be displayed immediately (box not checked), then the chosen storage path and filename will be displayed (see Figure 117).

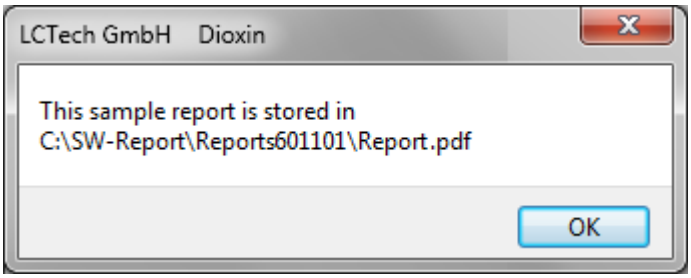


Figure 117: Display of the storage path for the generated report.

3) Button that creates the report in PDF format.

INFORMATION: Should the report to be produced already exist, then a message (Figure 114) will be displayed. Now, a new name or another sample file can be chosen.

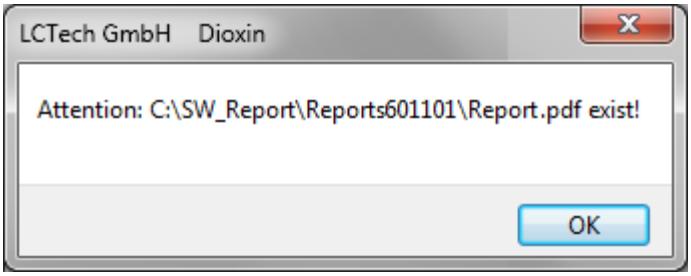


Figure 118: File duplicate notification.

4) Display of the selected sample file.

Pressing the button to specify the storage path will open a standard Windows dialog box as shown in Figure 119. Here, a file name can be chosen for the report to be produced. The selected name is accepted by pressing the "*Save*" button. Alternatively, pressing the "*Cancel*" button will abort renaming. However, a different storage path may be selected.

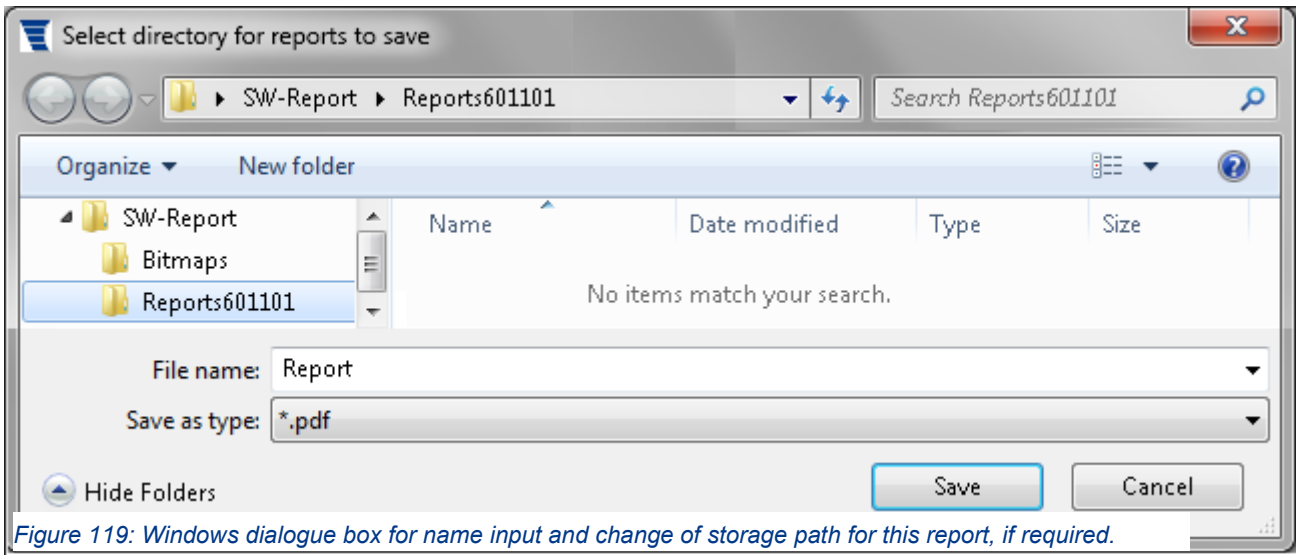


Figure 119: Windows dialogue box for name input and change of storage path for this report, if required.

If the suggested file name is not changed, then the final report will carry the same name as the selected sample file. Pressing the "*Create Report*" button will save the report in the current directory (the software installation directory) in an automatically created folder named *Report*, followed by the six-digit serial number of the DEXTech Heat device (see Figure 120, highlighted in grey). When selecting a), all future reports, which are saved in a PDF file, will be collected in this folder.

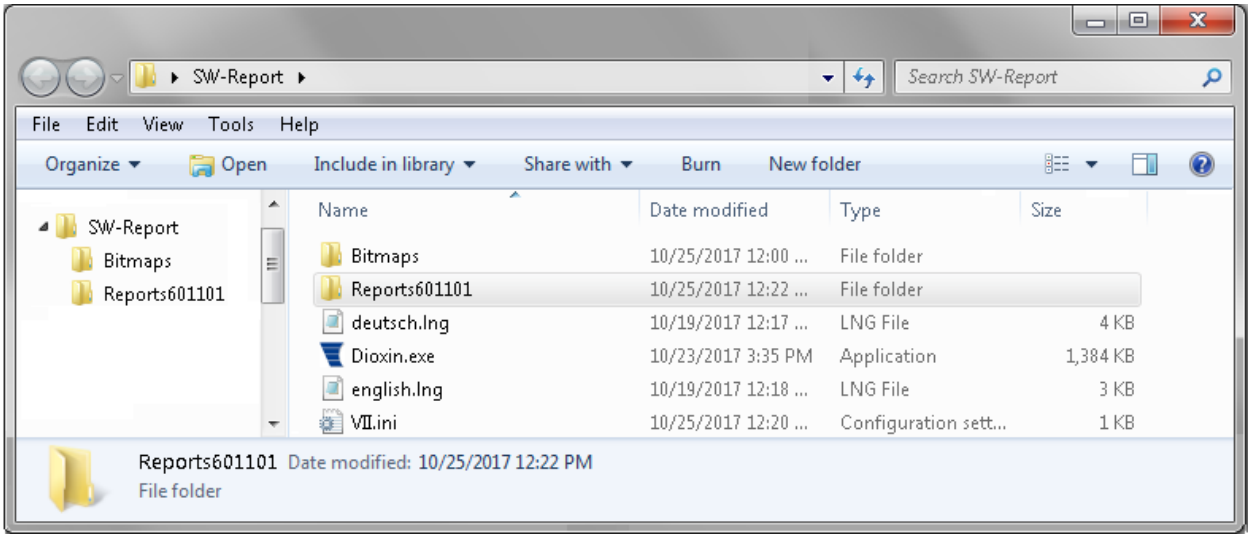


Figure 120: Current directory of the installation software now includes the folder "Reports601101" (highlighted in grey), which will hold all future reports of the respective DEXTech Heat device.

Option b) individually in PDF files:

Ticking "*selected data*" lists all stored sample data individually with their corresponding sample name (see Figure 121). By checking the individual sample data, specific sample names can be selected for reporting.

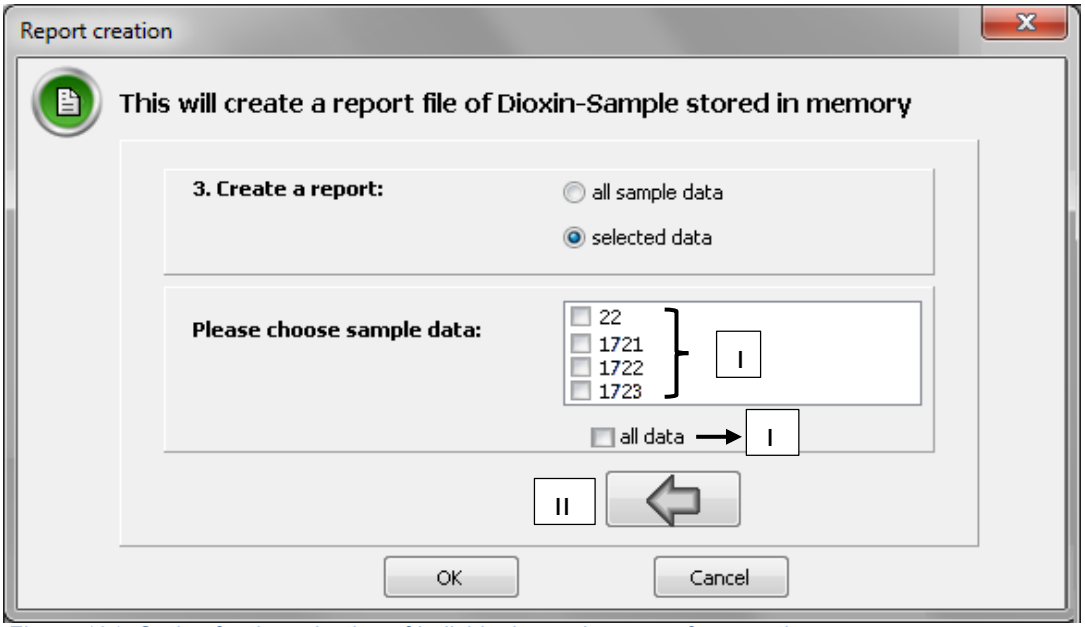


Figure 121: Option for the selection of individual sample names for reporting.

I. Individual checking of the required sample names.

II. Select all sample names.

III. "*Back*" arrow to return to the previous page.

After your selection (see Figure 122), a "*Forward*" arrow will appear, which when clicked progresses to the next page.

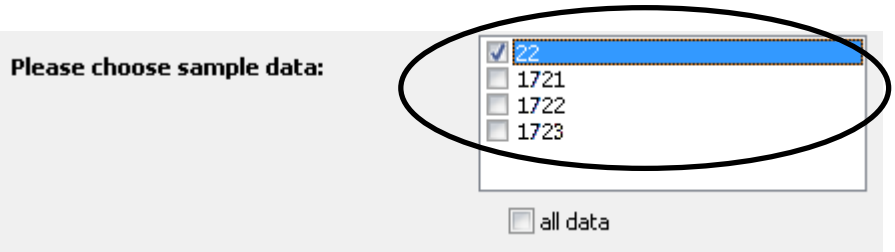


Figure 122: Selected sample data to be used for reporting.

This page completes the report generation (Figure 123).

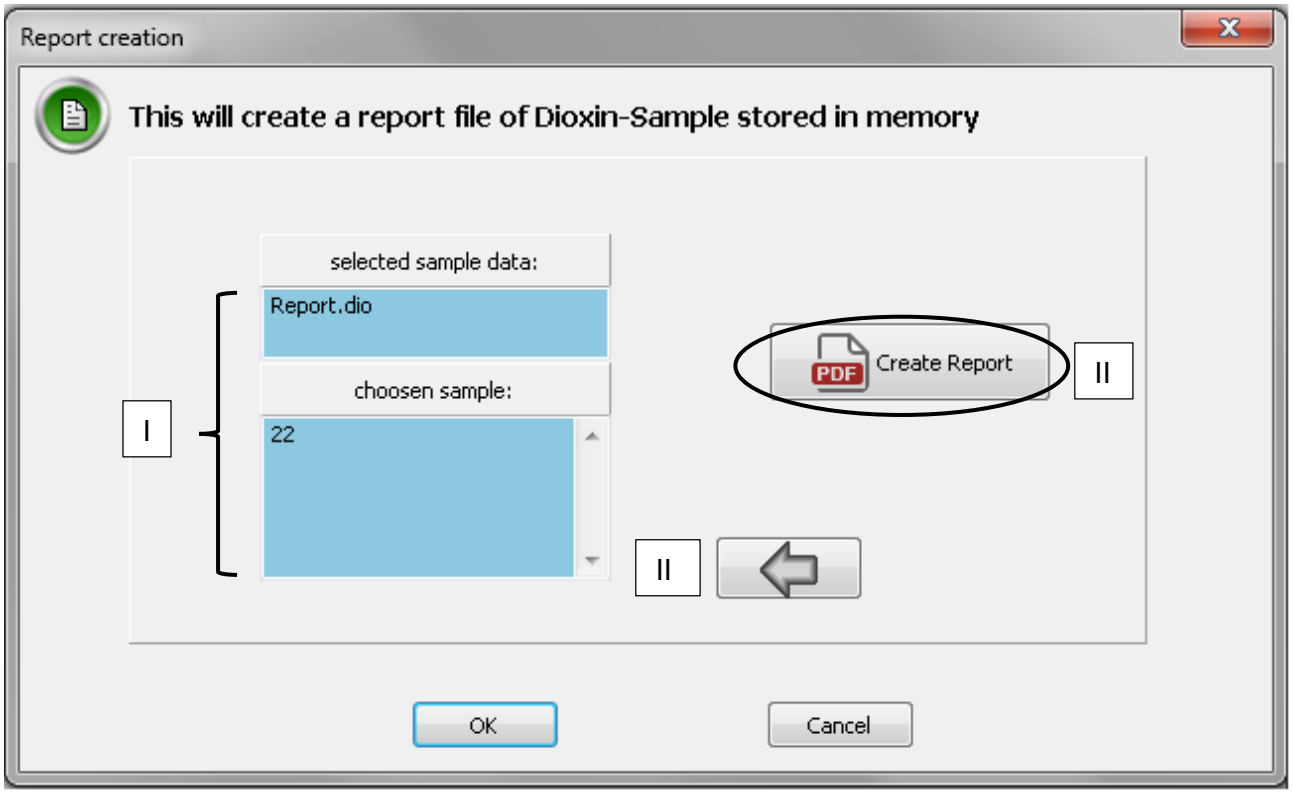


Figure 123: Final page with overview of the selected data and button to create the reports.

Overview of the sample file used and the selected samples from which a report is to be generated.

I. Button that concludes the report generation.

II. "*Back*" button if different samples need to be selected.

INFORMATION: This is only possible, if the "*Create Report*" button has not been clicked.

Important: After report generation, the respective sample data (for example Report.dio) will not be deleted automatically from the USB stick. These data must be specifically deleted by the user.

The reports will be stored in the current directory (the software installation directory). For this purpose, a folder named “Report”, followed by the six-digit serial number of the DEXTech Heat device, will be automatically created. For clarity, in this report folder another folder containing the current date will be included. The exact storage path of the reports is issued by a notification as shown in Figure 124.

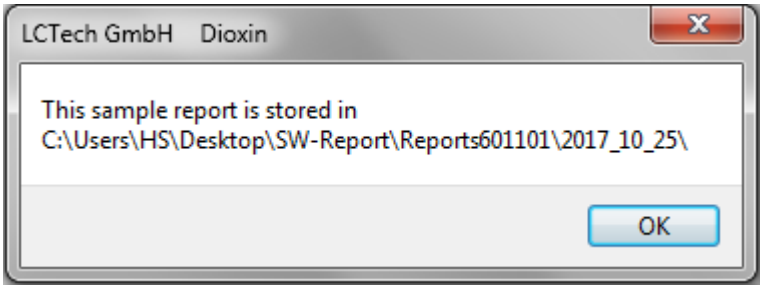


Figure 124: Issue of the storage path for the reports.

INFORMATION: If several individual reports are created in one day, then all reports will be saved in the same file (with the corresponding date and time, Figure 121).

Should a PDF file already exist on the system, automatically a new storage name is generated. For example: existing data 22.pdf → new name: 22_1.pdf.

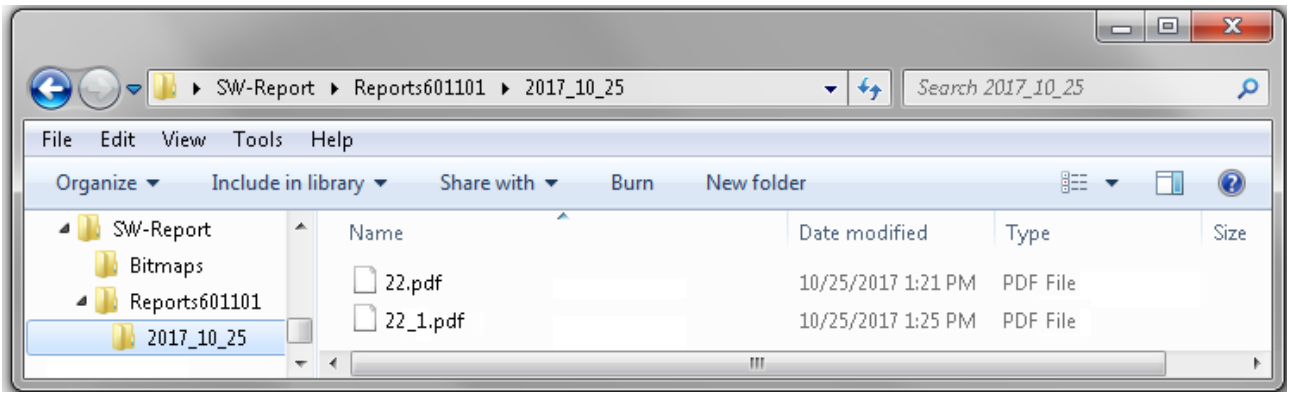


Figure 125: File with date (of the report creation) that contains all reports, which have been generated on this day.

Once the reporting is completed, the button "*New Report*" will appear (see Figure 126), with which the next report can be created.

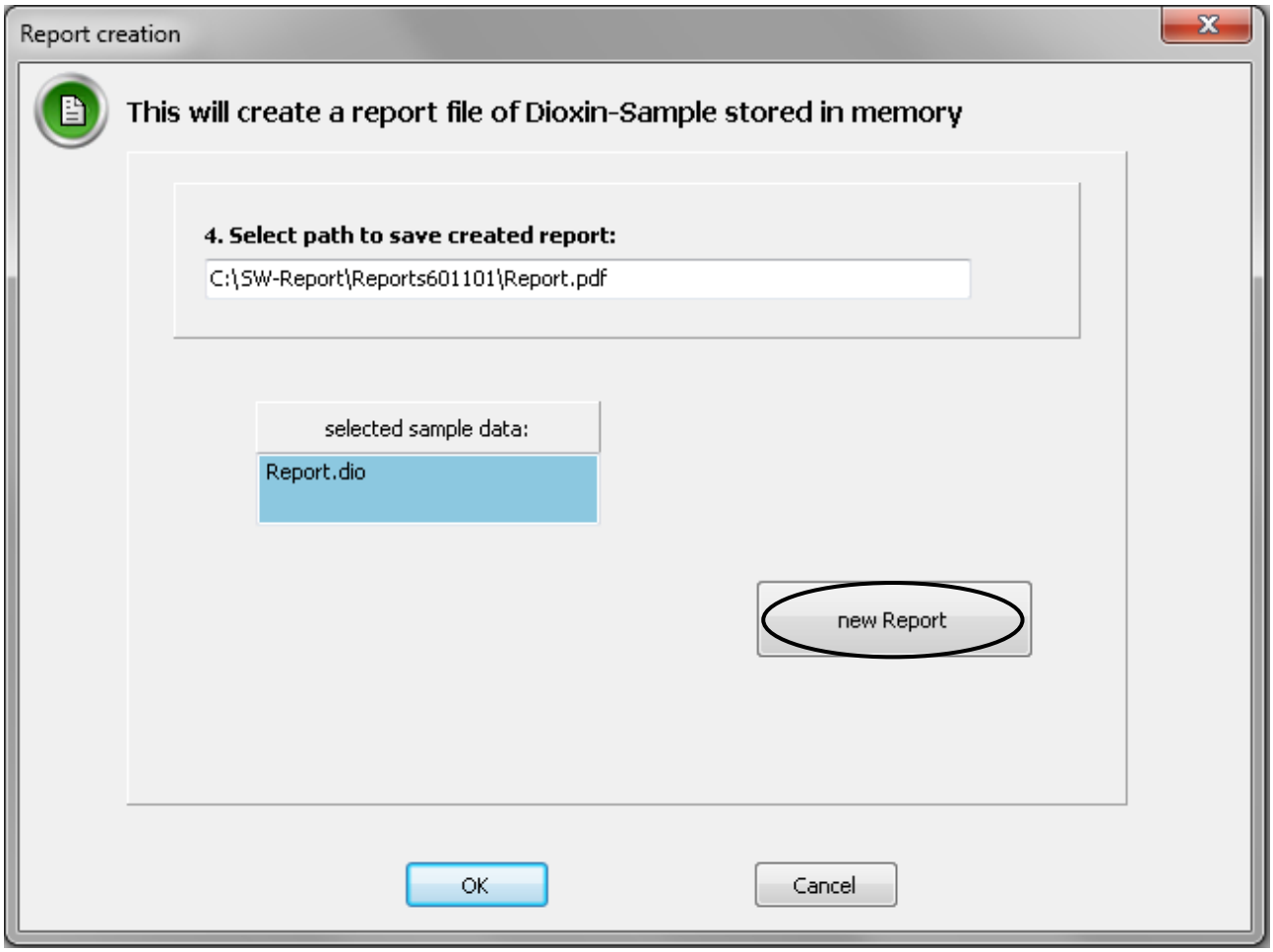


Figure 126: Finished report generation.

Report examples are shown in the following. Figure 127 illustrates a report without interruptions during a sample preparation.

Date: 10/1/2018 Time: 5:08:55 PM

DEXTech Heat

LCTech Dioxine - Sample report

Report name: C:\SW_Report\Reports601101\Report.pdf

Sample name:	22
Method name:	Alox Plus
Sample volume:	17 mL
User:	LCTech
Equipment-ID:	601101
Software version:	1.00
Sample loop volume:	20 ml
Maximum pressure:	15 bar
Maximum pressure Column 1:	5 bar
Heating state:	Off
Rinsed system:	Yes
Rinsed needle:	Yes
Round bottom flask wait time:	0.1 min
Start Time:	Mon Oct 1 14:48:27 2018
End Time:	Mon Oct 1 15:53:49 2018

Process steps:

Conditioning 1	Solvent: n-Hexane	Flow rate: 7 ml/min	Time: 15 min
Conditioning 2	Solvent: n-Hexane	Flow rate: 7 ml/min	Time: 0 min
Conditioning 3	Solvent: Toluene	Flow rate: 7 ml/min	Time: 0 min
Conditioning 4	Solvent: DCM/n-Hexane	Flow rate: 7 ml/min	Time: 0 min
Pre-run C1	Solvent: n-Hexane	Flow rate: 7 ml/min	Time: 3 min
Pre-run Fraction 1	Solvent: n-Hexane	Flow rate: 7 ml/min	Time: 26 min
Fraction 1	Solvent: DCM/n-Hexane	Flow rate: 3 ml/min	Time: 8 min
Fraction 2	Solvent: Toluene	Flow rate: 1 ml/min	Time: 10 min
Drying	Nitrogen		Time: 0 min

Rinsing steps:

Vial:	Step 1	Solvent: n-Hexane	Volume: 1 ml
	Step 2	Solvent: ---	Volume: 0 ml
	Step 3	Solvent: ---	Volume: 0 ml

Rinsing program:

Needle:	Exterior rinsing 1	Solvent: Toluene	Volume: 1 ml
	Exterior rinsing 2	Solvent: n-Hexane	Volume: 3 ml
	Interior rinsing 1	Solvent: Toluene	Volume: 1 ml
	Interior rinsing 2	Solvent: n-Hexane	Volume: 3 ml

Empty event list

www.LCTech.de , info@lctech.de

Figure 127: Example of a report without any notable events during sample preparation.

Report example with listed errors that occurred during sample preparation (Figure 128).

Date: 10/1/2018 Time: 5:08:55 PM

DEXTech Heat

LCTech Dioxine - Sample report

Report name: C:\SW_Report\Reports601101\1050.pdf

Sample name:	1050
Method name:	Alox Plus
Sample volume:	17 mL
User:	LCTech
Equipment-ID:	601101
Software version:	1.00
Sample loop volume:	20 ml
Maximum pressure:	15 bar
Maximum pressure Column 1:	5 bar
Heating state:	On
Rinsed system:	Yes
Rinsed needle:	Yes
Round bottom flask wait time:	0.0 min
Start Time:	Mon Oct 1 10:47:22 2018
End Time:	Mon Oct 1 12:00:15 2018

Process steps:

Conditioning 1	Solvent: n-Hexane	Flow rate: 7 ml/min	Time: 15 min
Conditioning 2	Solvent: n-Hexane	Flow rate: 7 ml/min	Time: 0 min
Conditioning 3	Solvent: Toluene	Flow rate: 7 ml/min	Time: 0 min
Conditioning 4	Solvent: DCM/n-Hexane	Flow rate: 7 ml/min	Time: 0 min
Pre-run C1	Solvent: n-Hexane	Flow rate: 7 ml/min	Time: 3 min
Pre-run Fraction 1	Solvent: n-Hexane	Flow rate: 7 ml/min	Time: 26 min
Fraction 1	Solvent: DCM/n-Hexane	Flow rate: 3 ml/min	Time: 8 min
Fraction 2	Solvent: Toluene	Flow rate: 1 ml/min	Time: 10 min
Drying	Nitrogen		Time: 0 min

Rinsing steps:

Vial:	Step 1	Solvent: n-Hexane	Volume: 1 ml
	Step 2	Solvent: ---	Volume: 0 ml
	Step 3	Solvent: ---	Volume: 0 ml

Rinsing program:

Needle:	Exterior rinsing 1	Solvent: Toluene	Volume: 1 ml
	Exterior rinsing 2	Solvent: n-Hexane	Volume: 3 ml
	Interior rinsing 1	Solvent: Toluene	Volume: 1 ml
	Interior rinsing 2	Solvent: n-Hexane	Volume: 3 ml

Event list:

Break	Time: Mon Oct 1 10:47:30 2018	Step: Conditioning 1	Duration: 0,1 min
Leakage	Time: Mon Oct 1 10:52:38 2018	Step: Pre-run Fraction C1	Duration: 0,3 min
Waste full	Time: Mon Oct 1 10:58:11 2018	Step: Pre-run Fraction C1	Duration: 8,5 min

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Figure 128: Example of a report with a list of errors. The error list contains all relevant information about the errors that occurred.

The report error list can contain a maximum of 15 errors. These include:

1) **Break**: During the process, the pause button has been pressed by the user.
2) **Leakage**: The leakage sensor has detected solvent in the tube trap of the drip tray.
3) **Waste full**: Error message, if the system is equipped with an optional float switch for the solvent waste. In this case, the sensor detects a signal at the waste flask.
4) **Overpressure**: The pressure value of the system is greater than 15 bar (217.7 psi).
5) **Overpressure column 1**: The pressure limit for the first column (Universal column) in the processing steps: Conditioning 1, Pre-run C1, Pre-run Fraction 1 or Pre-run Fraction 2 is greater than 5 bar (72.6 psi).
6) **Error IV**: Incorrect positioning of the injection valve.
7) **Error VV**: Error in positioning of the diverter valve.
8) **Error LsmV**: Position error of the solvent valve.
9) **Error ZV1**: Error in positioning of the central valve 1.
10) **Error ZV2**: The central valve 2 has not reached the desired position.
11) **Power failure**: The process was interrupted by a power failure.
12) **No process continuation**: After the power failure, the process was user-aborted and not continued.
13) **No process cancellation**: The user pressed the "*Cancel*" button, but then decided to continue.
14) **Process cancelled by user**: Process abortion by the user.
15) **Overpressure syringe pump:** Syringe pump failure through overpressure.
16) **Error syringe pump:** Timeout of syringe pump.
17) **Error initialization SP:** The syringe pump was not initialized during the booting process.
18) **Error Init SP:** The syringe pump cannot be initialized.
19) **Error pump:** The piston pump does not react to commands.
20) **SP valve error:** Syringe pump valve overload during the run.
21) **SP plunger error:** The plunger of the syringe pump does not react.

With each error notification, the following items are also documented:

- **Time of error**: Point of time at which the process was interrupted.

  → For example: Time: Mon Oct 23 10:38:20 2017

- **Step**: Current step before the interruption occurred.

  → For example: Step: Conditioning 1

- **Duration of interruption**: How long the interruption lasted.

  → For example: Duration: 0.8 min

Detailed information for specifics errors:

➔ 5) Change maximum pressure column 1.

Overpressure C1	Time: Mon Oct 23 10:38:20 2017	Step:Pre-run Fraction 1	Duration: 0.8 min
	Change maximum pressure Column 1: 6 bar	=> Attention: Pressure value out of specified range!	

➔ 12) Point of time of system restart.

Power blackout	Power fail restart Mon Oct 23 10:38:20 2017	Step: Conditioning 1

➔ 15) Point of time of abortion by user with details as to how long this step was active (ACTUAL duration) and how long this step should have been running (SET duration).

Process cancelled by user	Time: Mon Oct 23 10:38:20 2017	Step: Conditioning 3	at time: 1/5

ACTUAL SET

8. Set-up

DEXTech Heat is designed for two different configurations:

a) Alumina Plus

b) Alumina Pure

c) Dioxin only

Changes of the set-up (Figure 129) are possible after the start screen appears or in the main menu (see Figure 3).

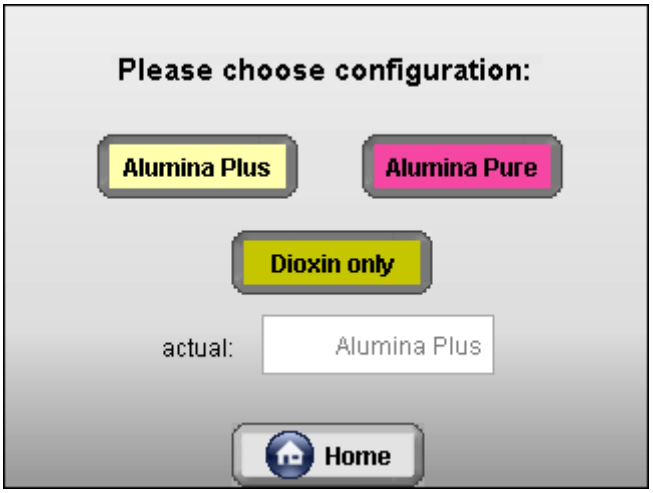


Figure 129: Set-up selection.

Changing the set-up causes different fractions.

a) Alumina Plus:

- Fraction 1: Fraction 1 only with column 1 (optional)
- Fraction 1: ndl PCB (Indicators), mono-ortho PCB, PBDEs
- Fraction 2: non-ortho PCB, PCDD/F

b) Alumina Pure:

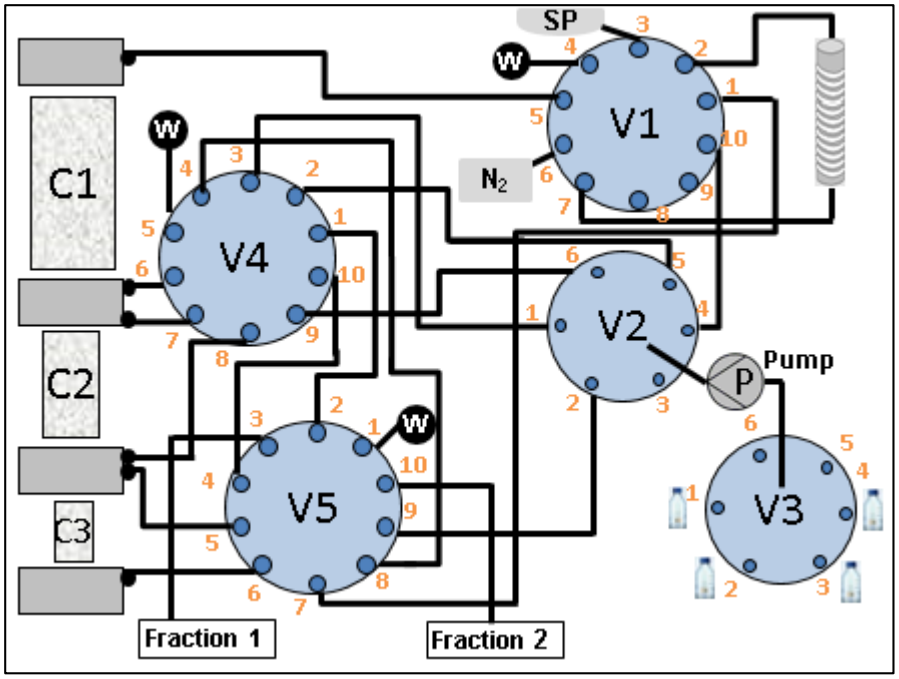
- Fraction 1:: Fraction 1 only with column 1 (optional)
- Fraction 1: ndl PCB (Indicators), mono-ortho PCB, non-ortho PCB, PBDEs
- Fraction 2: PCDD/F
- Fr

c) Dioxin only:

- Fraction 2: PCDD/F

9. Fluidics

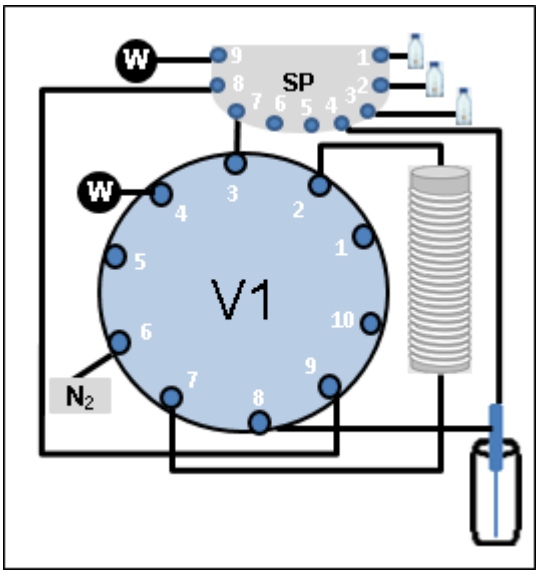
Below is an overview of the DEXTech Heat flow scheme.



V1: Injection valve
V2: Distribution valve
V3: Solvent valve
V4: Central valve 1
V5: Central valve 2
C1: Column 1 (Universal/SMART column)
C2: Column 2 (Alumina column)
C3: Column 3 (Carbon column)
W: Waste
N_2: Nitrogen

Flow scheme 1: Tubing plan of the DEXTech Heat.

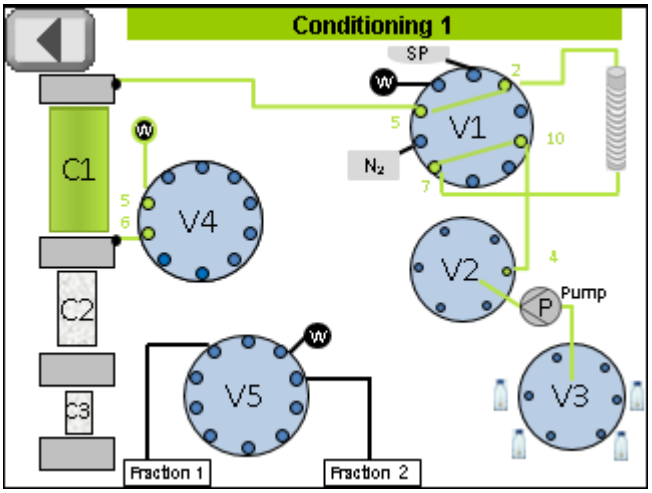
A more detailed view of the syringe pump tubing is illustrated in Flow scheme 2.



SP: Syringe pump
V1: (IV) Injection valve
W: Waste
N_2: Nitrogen

Flow scheme 2: Syringe pump.

For more detailed information, please refer to the software where the exact valve positioning can be seen. The relevant flow scheme will be displayed by either pressing the respective step in the method section or in the processing window (see section 5.3.6). An example diagram is shown in the following using the conditioning step 1.



Flow scheme 3: Conditioning 1.

10. Service

10.1. Emptying the Drip Tray

In case of a leak developing from tubings, columns or valves, a drip tray has been fitted at the bottom of the device (Figure 130, left), which is equipped with a solvent sensor (Figure 130, right).

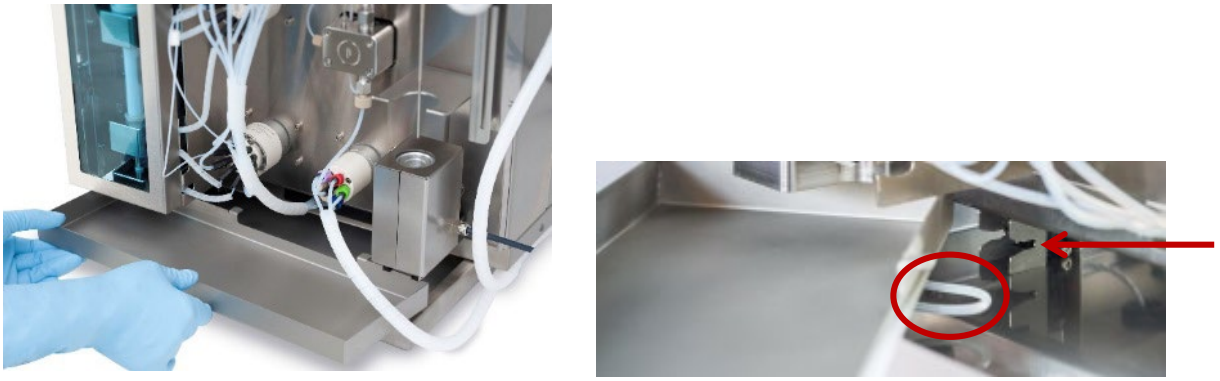


Figure 130: Left: removable drip tray; right: solvent sensor (red arrow) for solvent detection with a tube trap (circled in red).

If solvent is caught in the drip tray, it will run into the tube trap (Figure 130, right) and is detected there by the sensor. On detection, the system will stop and show the following error message on the display.

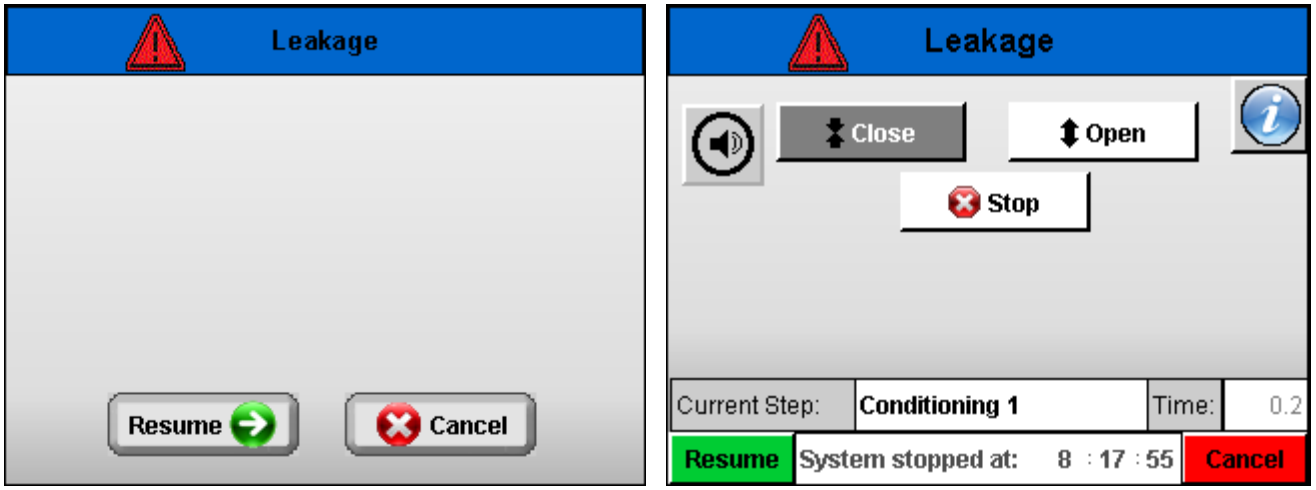


Figure 131: Leakage error. Left: error occurred during system bleeding and rinsing steps. Right: during processing.

To resume processing, the cause for the leakage needs to be found and rectified. As part of the fault finding procedure, all tubes must be checked for leaks to ascertain where the system loses the solvent. Check the connection points of the tubes on the valve heads. Next, examine the columns for leaks. Using a cloth may help identify the origin of the leak.

Caution: If a leak occurs, solvent can escape in an uncontrolled manner. Please always use hand and eye protection!

Once the leak has been found and remedied, the drip tray needs to be emptied in order for the operation to proceed.

Procedure for emptying the drip tray:

- Open the front door of the device (Figure 132).

Figure 132: Device with front door open.

- Detach the fraction rack (optional), remove the drip tray (see Figure 133) and remount the fraction rack. Generally, please pay special attention to the fraction tubes and the solvent supply. Then, empty the drip tray.

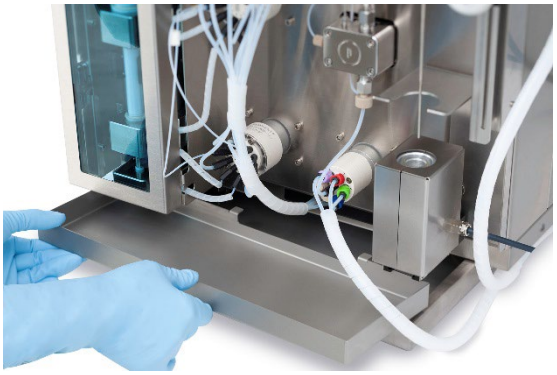


Figure 133: Removal of the drip tray.

NOTE: It is important to keep the tube trap clean and solvent-free. For best results, blow out with N_2.

- After the drip tray has been emptied, it must be replaced accurately to ensure the correct working of the solvent detection sensor.

ATTENTION: When inserting the drip tray, please observe that the tube is guided correctly within the sensor.

- This section of tubing is inserted into the recess (Figure 134) of the sensor.

Figure 134: Insertion of the tube trap.

- Finally, remount the fraction rack (optional) and close the front door.

10.2. Maintenance Recommendations: Syringe Pump

The piston of the syringe should be replaced in the event of a leakage. For more information on how to replace the syringe of the syringe pump, please contact service@LCTech.de.

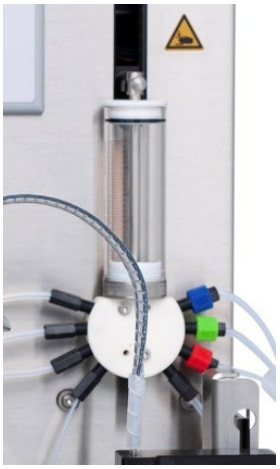


Figure 135: Syringe pump.

10.3. Software Maintenance

10.3.1 Internal Area

The software includes an internal area, which is password-protected (level 2 Chapter 5.6.2).

The 

- button appears after entering the password, shown in Figure 136.

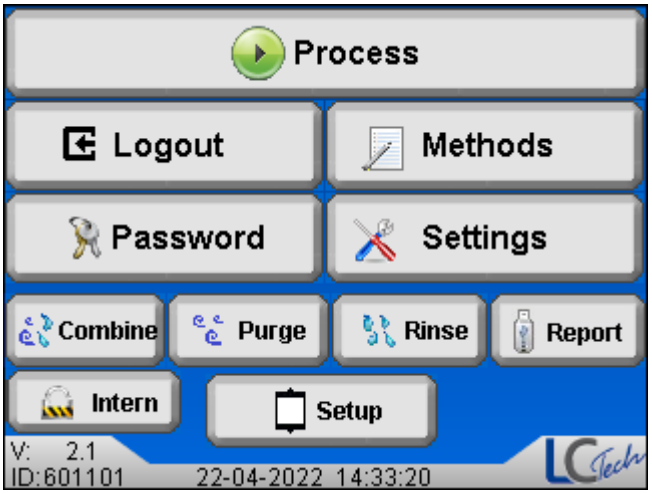


Figure 136: Homepage with internal button.

ATTENTION: Improper and arbitrary parameter change in the internal area can cause an error message and thus lead to malfunctioning of the system. The parameters should only be amended, where absolutely necessary.

Overview Page

Clicking on the "*Internal*" button leads to an overview page with a number of buttons (see Figure 137), each of which links to other pages.

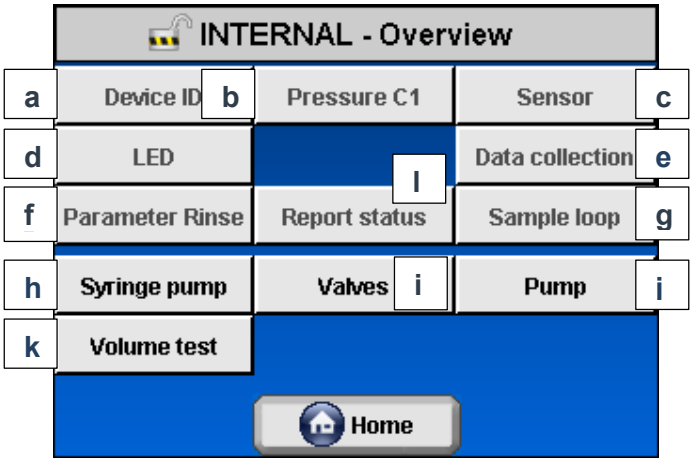


Figure 137: Internal area with various buttons labelled a –k.

a) *Device ID*: Input of device-ID.
b) *Pressure C1*: Set maximum pressure permanently for column 1.
c) *Sensor*: Status display of sensors and N_2-valve test.

d) *LED*: Test for colour coding LED.
e) *Data collection*: Display of operating hours and number of current sample processing.
f) *Parameter rinse*: Input rinse program of needle.
g) *Sample loop*: Definition of size and set injection parameter.
h) *Syringe pump*: Function test of syringe pump.
i) *Valve*: Switching option for all valves.
j) *Pump*: Test options for pump.
k) *Volume test*: Set the pump feed rate.

(a) Device ID

On the settings page (Figure 138), the device ID is entered.

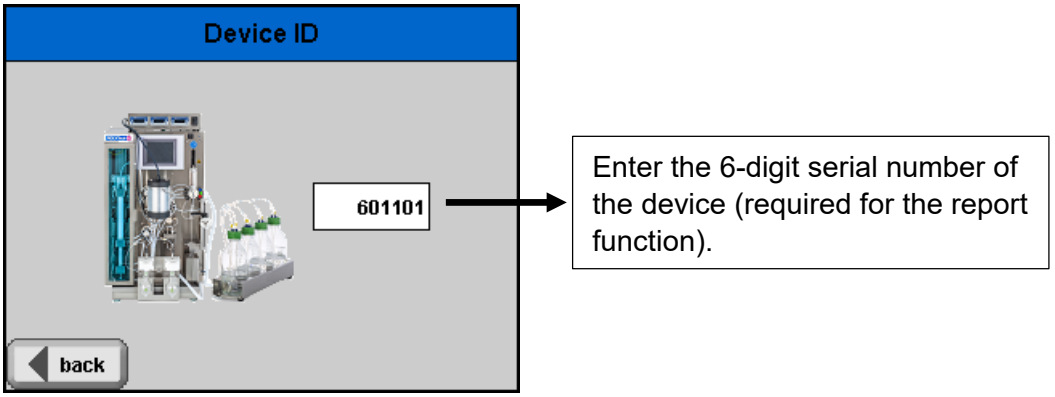


Figure 138: Device ID.

(b) Pressure C1

In this area, the maximum pressure can be set for column 1 (see Figure 139) and permanently stored on the memory-card.

NOTE: The set value is only considered in processing steps during the process while column 1 is in use. This depends on set-ups in the processing steps: “Conditioning 1”, “Pre-run C1”, “Pre-run fraction 1” and “Pre-run Fraction 2”.

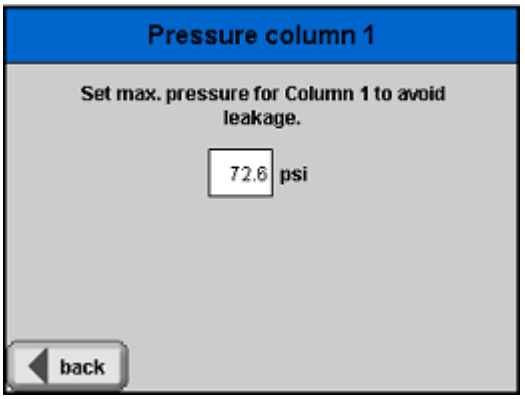
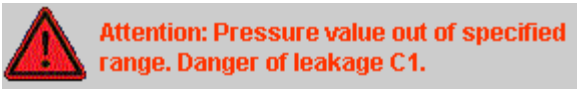


Figure 139: Setting maximum pressure of column 1.

The pressure value should not be set above 5.0 bar (72.6 psi). However, if a value is sethigher than 5.0 bar, the following safety message will appear.



NOTE: Only in the internal area, the pressure control can be stored permanently on the CF-card.

Example: Pre-set pressure in the internal area – max. pressure C1 is 5.0 bar. An error occurs in the process “Overpressure C1“ and subsequently, the pressure for column 1 is set to 5.8 bar in the error window. This "new" set pressure value for column 1 is only valid during the current process. The value stored on the CF-card remains at 5.0 bar.

→ In the event that a power failure occurred and the pressure value for column 1 had been re-set in the current process (before power failure), the newly assigned pressure value that was assigned before the power failure will still apply when the process resumes.

(c) Sensor

The subsequent sensor page serves the testing of the DEXTech Heat sensor functionality.

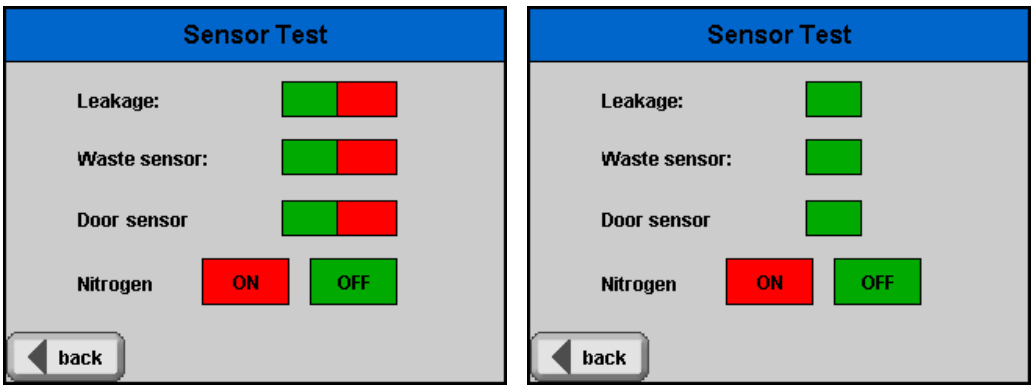


Figure 140: Sensor page with indication of the status of the respective sensor, the number of all previous sample preparations and of the operating hours.

➔ Depending on the status, either the green or the red box will be displayed.

Leakage sensor:	green = no solvent in the tube red = solvent in the tube
Waste sensor:	green = waste not full yet red = waste full
Nitrogen:	green “*OFF*“ = Nitrogen off red “*ON*“ = Valve on → N_2 on
Door sensor:	green = Door closed red = Door open

(d) LED

Figure 141 shows the control function for the LED status light. Depending on which button was pressed, the LED will light up in the colour of the corresponding number code.

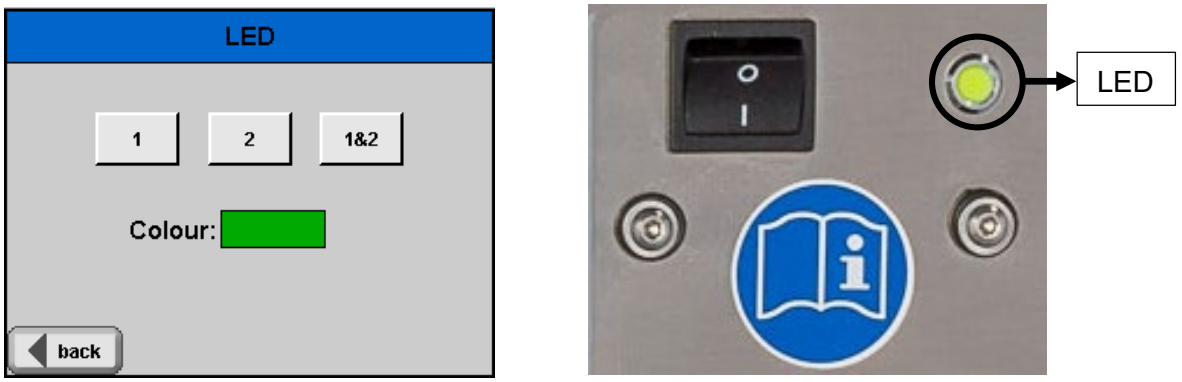


Figure 141: Left: control function for LED and right: position of the LED on the device.

Colour coding:

1 = 2 = 1&2 =

(e) Data collection

In this section (Figure 142), a counter for the number of sample preparations and pump operating hours is included.

NOTE: If conditioning and fractionation are carried out in separate methods, each event will cause a counter increment.

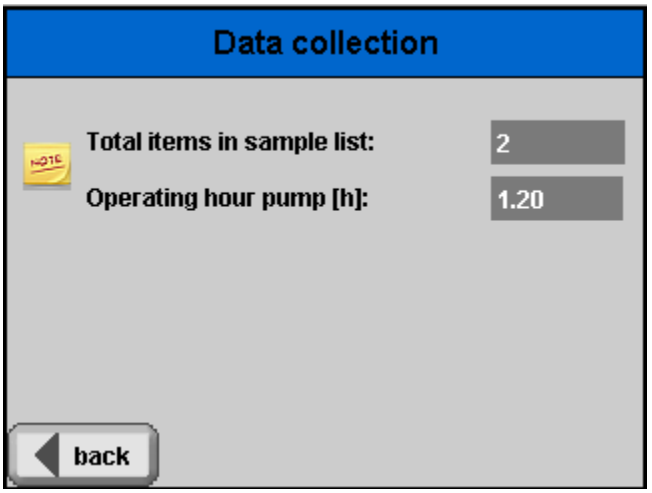


Figure 142: Sensor page with the number of all previous sample preparations and operating hours.

(f) Parameter rinse

Parameterisation options for the needle rinse, specified in Figure 143.

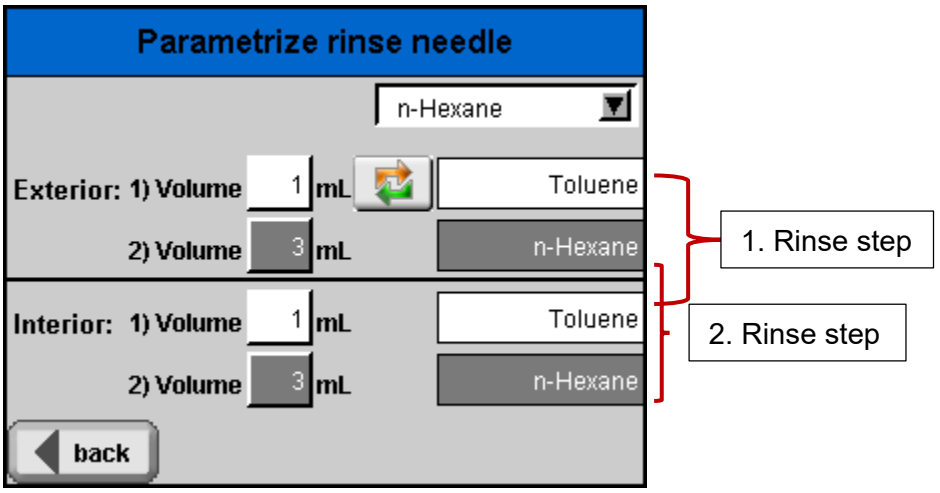


Figure 143: Needle rinse.

The rinsing of the sample needle consists of two steps. Each step includes the inside and outside cleaning of the needle. For each step one solvent only can be chosen.

INFORMATION: If the solvent in rinsing step 1 was not n-Hexane, then the solvent applied in the second step will be automatically n-Hexane.

(g) Sample loop

In this screen, you can select the size of the sample loop.

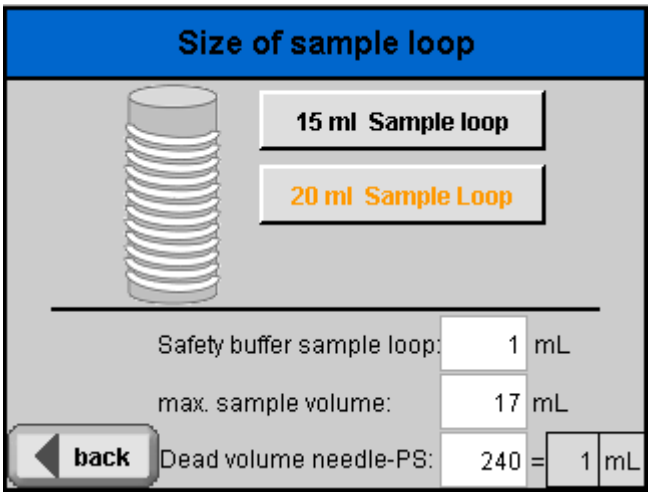


Figure 144: Sample loop.

INFORMATION: If the volume of the sample loop is changed, care has to be taken that the max. sample volume and the rinsing volume of the sample loop have to be changed too. The corresponding data and volumes can be requested from our LCTech service team .

Volumes can be chosen for the following areas:

Safety buffer sample loop*:* This value represents a safety factor to prevent overloading of the sample loop. This value also defines the maximum sample volume

sample volume - safety buffer sample loop = max sample volume

Example: 20 mL - 1 mL = 19 mL

Max. sample volume*:* This is the maximum sample volume that can be chosen for each method in the Method section (see Chapter 5.4 Menu Method→ Load sample → Figure 31).

Dead volume needle-PS: This volume defines the dead volume of the tubing from the sample loop to the needle.

(h) Syringe pump

In this window, different syringe pump functions can be controlled (Figure 145).

INIT: Piston and valve move to the Init position.

Settings: Set parameters are displayed.

Service: Piston moves to the service position.

Info button: The actual position of the valve and the piston are shown.

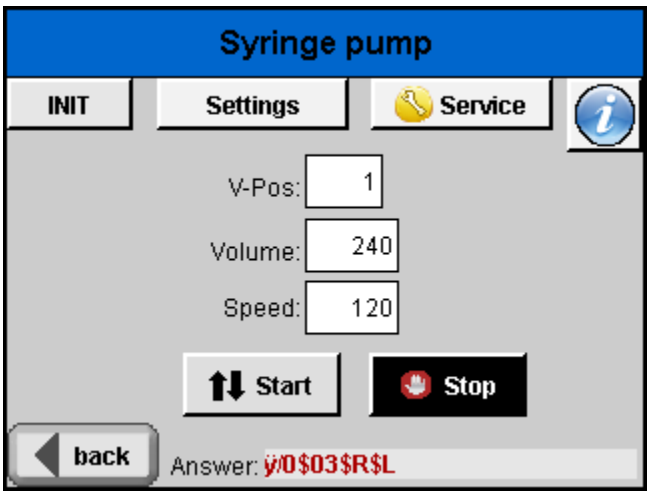


Figure 145: Syringe pump test window.

(i) Valve

The valve page offers the option to switch the individual valves separately to different positions (see Figure 146).

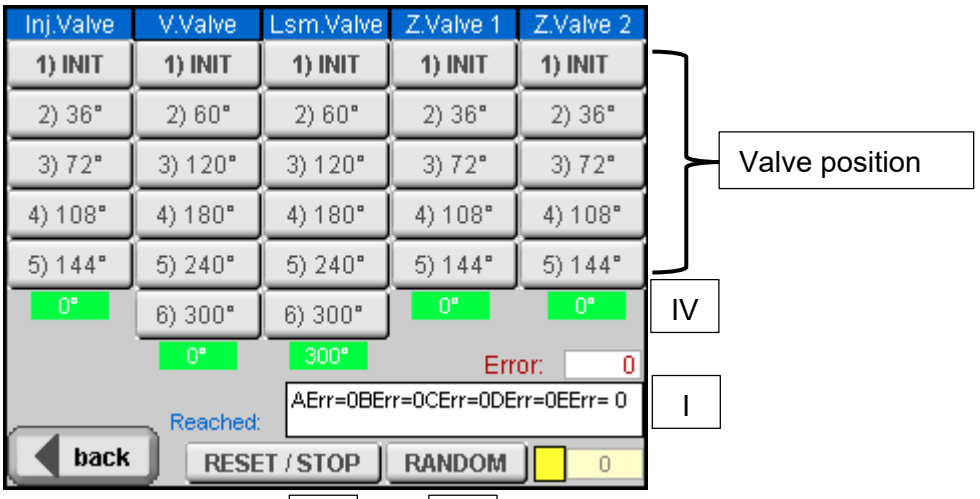


Figure 146: Valve page.

I. Continuous display of the position and potential error state of the valve.

II. Simultaneous reset function for all valves. The random mode will be stopped.

III. The random mode switches all five valves simultaneously to different and random positions at intervals of ca. 1 minute (the "*RESET/STOP*" button ends this mode).

IV. Current valve position for each valve. If a valve has a positioning error, the background colour changes from green to red. An example is shown in Figure 147.

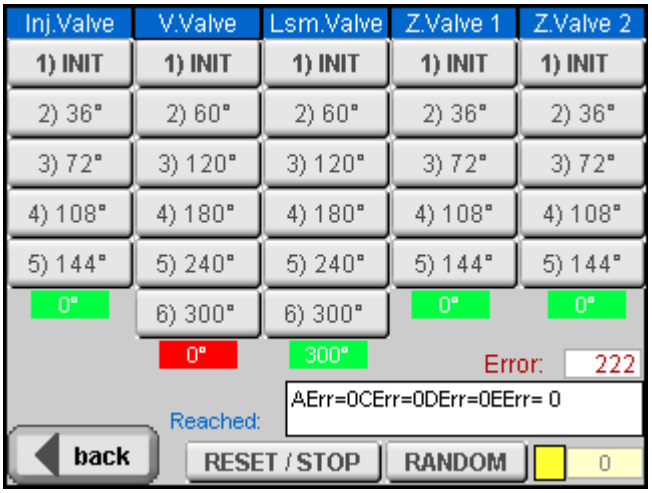


Figure 147: Valve position with error signaling.

INFORMATION: If you leave this window, the solvent valve (Lsm.Valve) will automatically move to the 300° blocked position to prevent backflow of the solvent.

(j) Pump

In this window, the flow rate of the pump can be tested with different solvents (Figure 148), and different flow rates can be combined with different times (pump duration). During the test, the system pressure is monitored and shown in the window. Additionally, the pressure sensor can be calibrated.

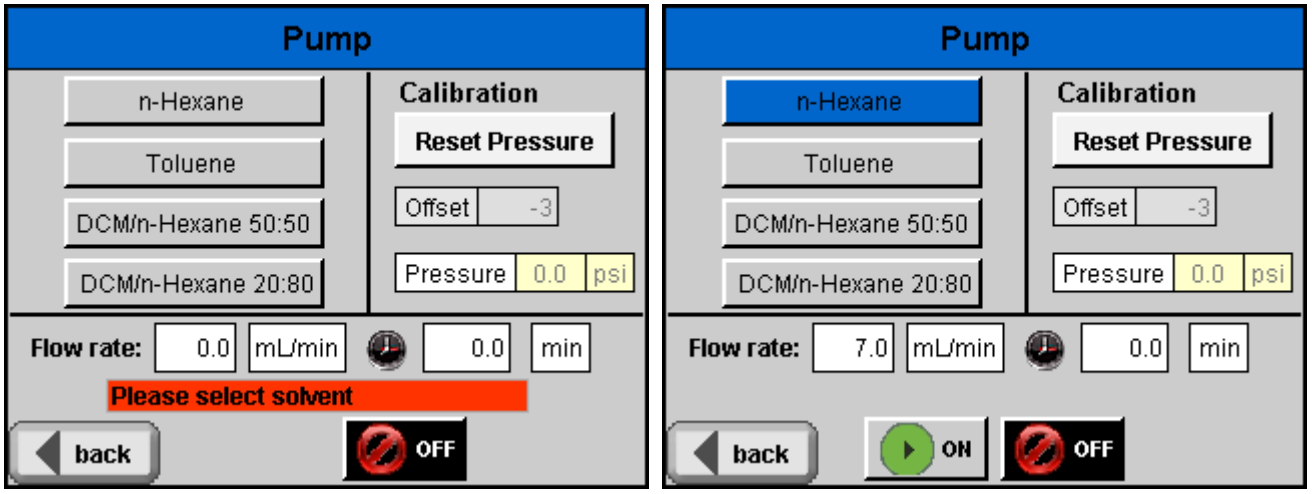


Figure 148: Testing the delivery volume of the pump.

For safety, a pressure indicator (Figure 149) is added, which stops the pump immediately, if the stored maximum pressure value is exceeded. Within the error message box, the maximum pressure can be adjusted as required.

Figure 149: Pressure indicator contained within the volume test.

NOTE: The maximum pressure is limited to 15 bar (217.7 psi).

(k) Volume test

Option to control the pump flow rate. Hold fraction 2 (Figure 150) into a measuring cylinder.

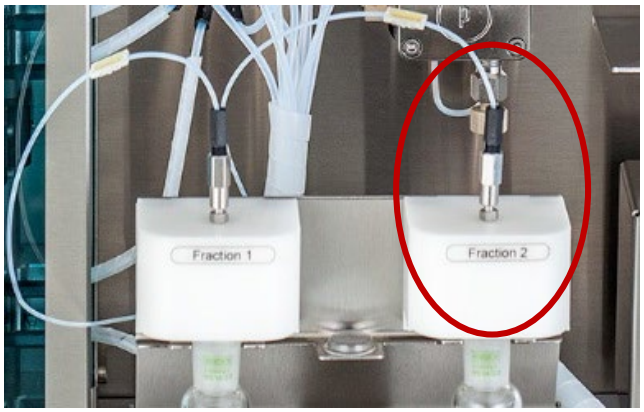


Figure 150: Volume test using fraction 2.

Then, set the parameters solvent "*n-Hexane*", flow rate, and time (i.e. how long should the pump deliver for) in the software, as illustrated in the figure below.

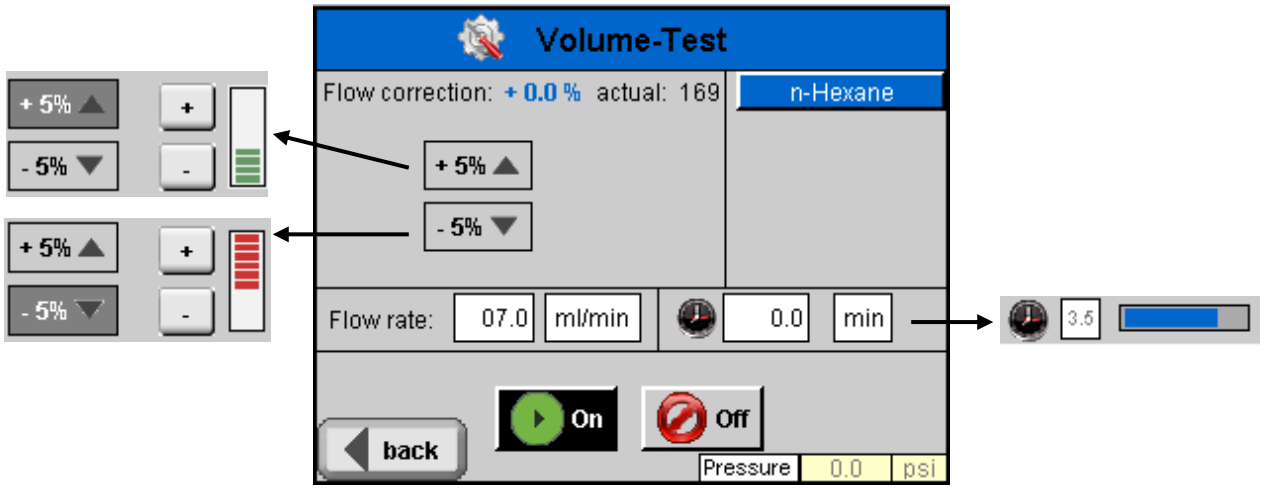


Figure 151: Testing the delivery volume of the pump. For this purpose, choose solvent "n-Hexane", enter a flow rate (for example 7 mL/min), and a period of time (for example 5 minutes).

Should the end volume not correspond to the required value, a flow correction by +/- 5 % can be undertaken through adjusting the pump speed.

NOTE: The adjustment of the pump speed has a range from 160 to 177. The default value is set to 169.

At the end of the volume test (when the set time has elapsed and the pump is switched off), a query window will appear (Figure 152) with the option to accept and save the set value. Clicking "*Yes*" accepts the value, which is then stored in the Ini-file.

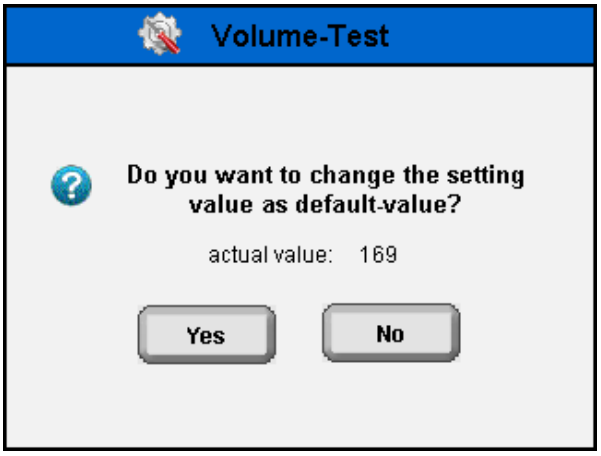


Figure 152: Query window with function to accept the set value.

In addition, pressure supervision takes place at this point. An overpressure situation (i. e. pressure > maximum pressure value) will stop the pump immediately and an overpressure announcement (Figure 149) will be displayed on the DEXTech Heat.

(l) Report status

Feature to enable and disable the report function (Figure 153). The grey „*ON*" button displays an active report function. The sample counter shows the number of already recorded sample preparations.

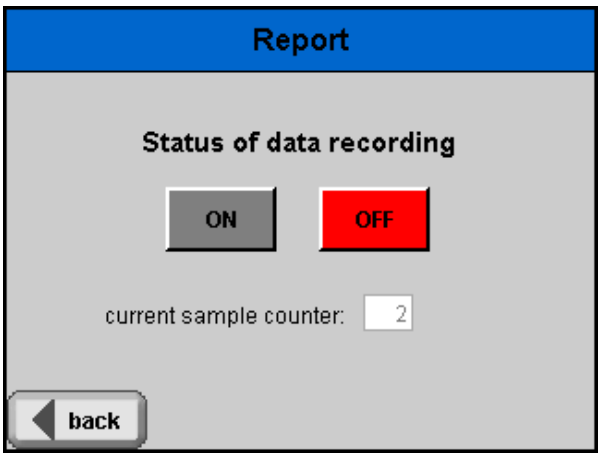


Figure 153: Status of report function.

In case of deactivated report data logging („*OFF*“ button) a warning as shown in Figure 154 will appear. A confirmation of the warning “Sample data will be deleted” with the “Yes” button results in deactivated data recording and deletes all existing report data. Pressing the “No” button do not change the report status.

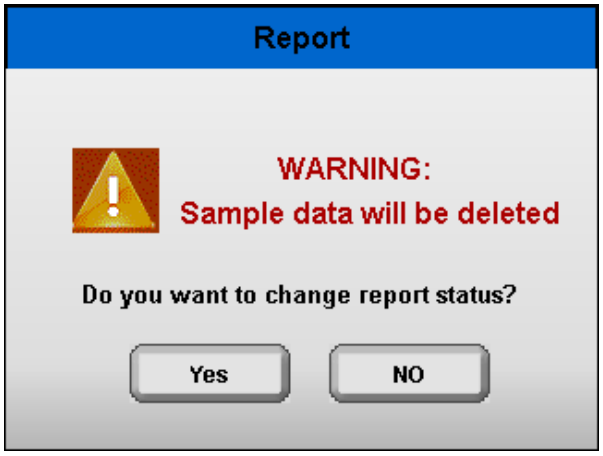


Figure 154: Warning before report data are deleted.

10.3.2 Service Area

The service area contains a status display of relevant parts as well as maintenance recommendations for wear parts of the pump and the five valves.

In the main window, a red service button will appear if an error on one of the relevant parts arises or if the wear parts of the pump or the valves reach a certain number (e.g. pump running time, valve circuits).

If this button appears, the status of certain parts is not correct or a corresponding maintenance step needs to be carried out. To locate the service part, go to "*Settings*" → “*Service*”, where you will find an accurate list of all serviceable parts. An example is shown in the following figure.

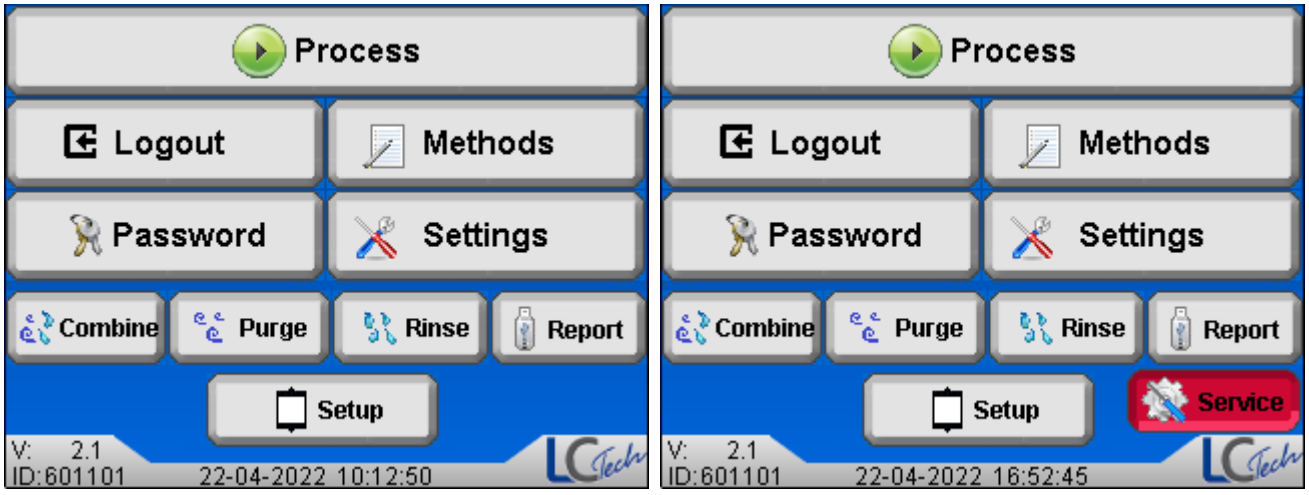


Figure 155: Main window without (left) and with (right) service display.

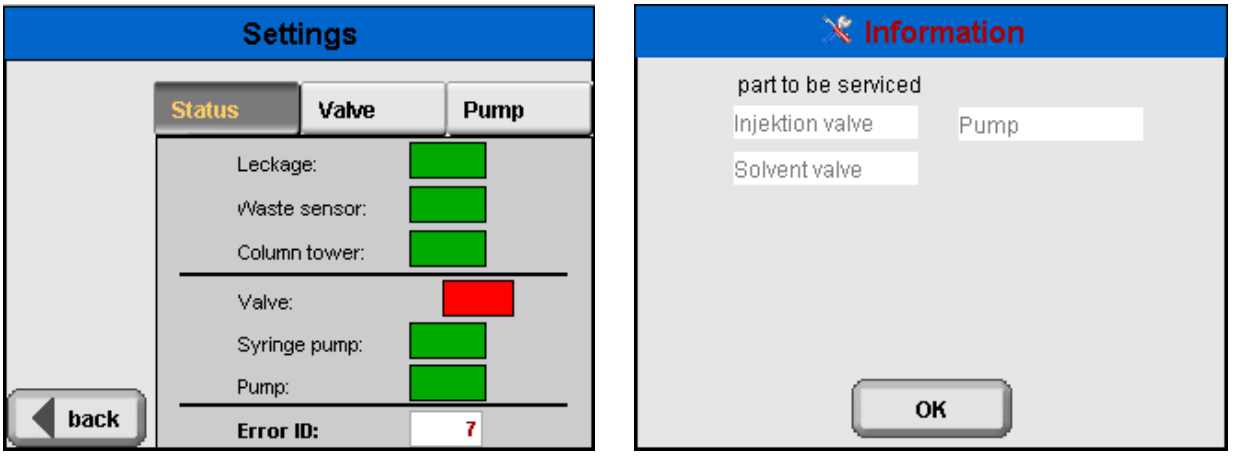


Figure 156: Service window with display the status of relevant parts (left) and of all serviceable parts (right).

For the determination of the wearing part (depending on the information note), the maintenance settings for valves/pump need to be reviewed (see Figure 157).

NOTE: Parameter setting is only possible with prior entry of the password. Otherwise, the input fields will be locked: 123

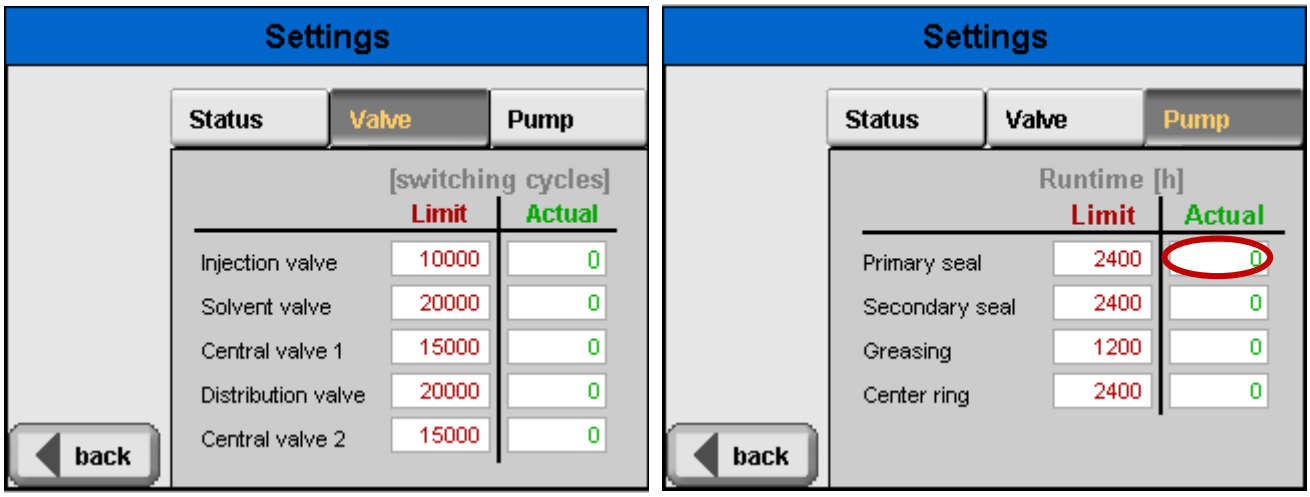


Figure 157: Settings page for maintenance of valves (left) and pump (right).

A service is required, when the "*Limit*" point (corresponds to the recommended value when a component needs to be replaced) is exceeded. That is, the "*Actual*" value (the current value of the component) is greater than the set point.

For the maintenance of pump or valve, the respective component has to be changed. The components of the pump are listed separately and the rotor seal of the corresponding valve needs to be renewed. Then the "*Actual*" value of the serviced part has to be reset to 0 (Figure 157, marked in red on the right), and the service message will disappear from the main window.

NOTE: For general disabling of this service function, set the "*Limit*" value to 0. Thus, no service message will appear in the main window. The "*Actual*" value will still be counted.

10.4. Replacement Tubes

The DEXTech Heat system uses 2 types of tubing. For suction tubes and sample loop, PTFE tubes are used with a ⅛" OD and a 1/16" ID. Sample loop has also a PTFE tube with a diameter of 3 x 2 mm. All remaining tubes are made of PTFE with a 1/16" OD and 0.75 mm ID.

Valve and block screw connections have a UNF ¼"-28 thread. At the pump head and pressure sensor, the end of the bore holes is tapered.

INFORMATION: To make the sealing joints, the following items are recommended: DEXTech Heat First Aid Kit (Part-Number: 16039), this requires the Easy-Flange-Kit (Part-Number: 10517).

Two sealing systems are used for the connection of tubes with valves or blocks:

- The connections to the pump head and the pressure sensor are crimp connections with a cone.
- As to the connections to the blocks in the column tower and to the ends of the sample loop, the tube ends are flanged (flange connection) in order to prevent the press ring of a crimp connection getting caught on removal of the tube.

ATTENTION: When replacing tube connections, solvents may leak out. Please use hand protection.

The type of sealing system at the end of a tube depends on the destination of the respective tube and on what the tube will be connected to.

NOTE: Bent tubes must be completely replaced.

To restore a tube connection, the existing tube may be shortened once by a max. length of 10 mm.

10.4.1 Crimp Connection

Preparing a crimp connection in 1/16“ tubes for tapered bores

Materials: Finger-tight fitting

Procedure:

Cut the tube end square and slide the finger-tight fitting over the tube such that the tube protrudes from the fitting by ca. 5 mm on the bore side (Figure 158). Then screw the fitting hand-tight into the bore.

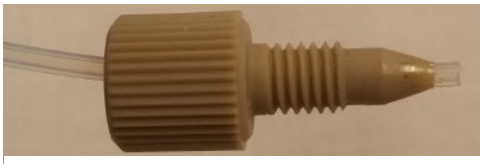


Figure 158: Mounting of the finger-tight fitting onto the tube.

ALTERNATIVE: Another helpful method to produce this connection is by means of a union connector that has a ¼“-28 as well as a tapered bore (Part-Number: 10419, Figure 159). By using a union connector with a tapered bore, it is possible to produce the connection outside of the system, which makes this process considerably easier.

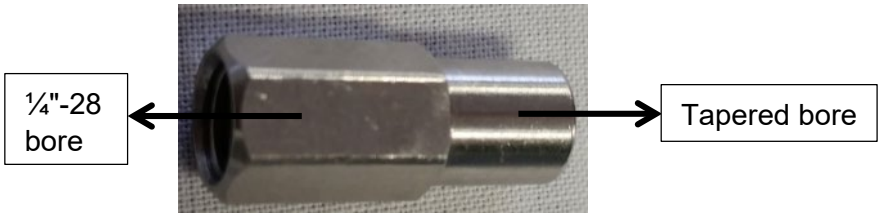


Figure 159: Union connector with two different types of bore.

Hand-tightening seals the connection, shown in Figure 160.

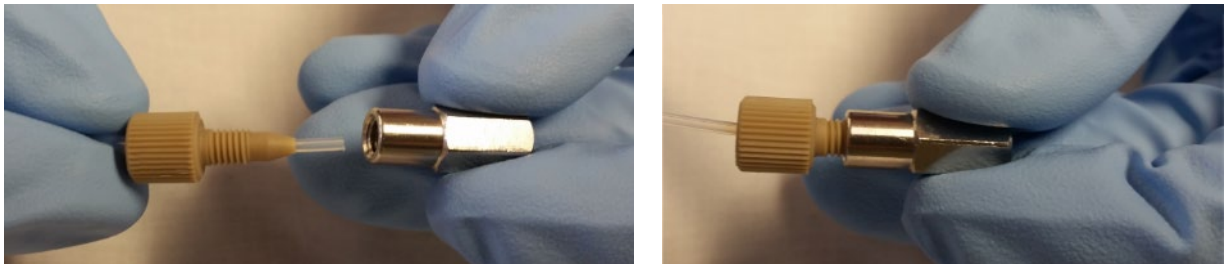


Figure 160: Producing a crimp connection for a tapered bore using a union connector.

10.4.2 Flange Connection

1) Preparing a flange connection with ⅛“ tubing

Materials: NUT ¼"-28 (Part-Number: 10027)

Ring (Part-Number: 10437)

Flanging Tools (Easy-Flange-Kit, Part-Number: 10517)

Optional: Colour Adapter (Part-Number: 10028-10039)

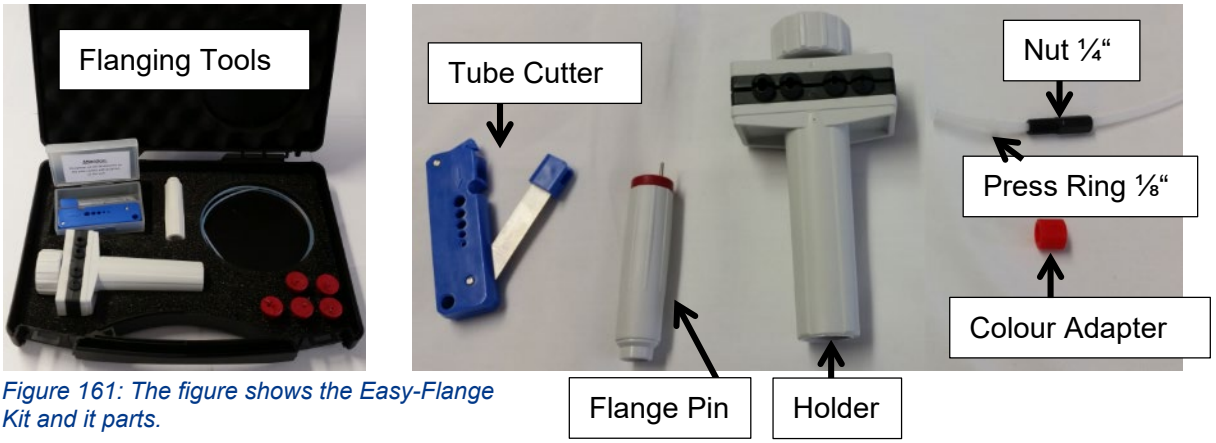


Figure 161: The figure shows the Easy-Flange Kit and it parts.

Procedure:

Cut the tube end square and slide the NUT ¼"-28 and ring onto the tube. Then insert the tube into the holder (all steps shown in Figure 162).

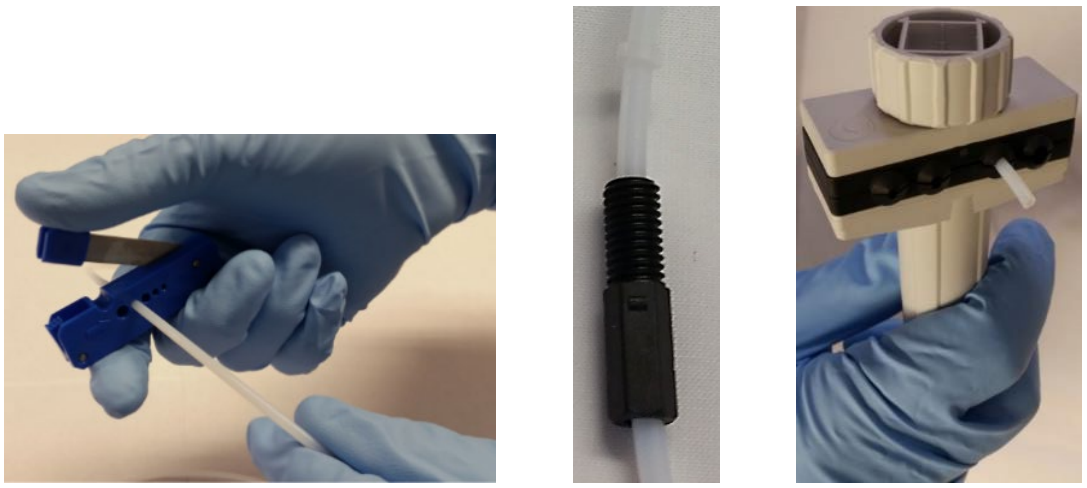


Figure 162: Preparation for flanging of the tube. Left cutting the tube end, centre assembling the materials and right insertion of the tube into the flanging holder.

With the hollow end of the flange pin, push the tube back in the direction of the holder in order to gain the correct projection required for the width of the flange. Then fix the tube in the holder. Put the 1.3 mm disc insert onto the flange pin and insert the pin into the tube hole (Figure 163, left). By rotating the flange pin and applying pressure in forward direction, the PTFE material is cold formed (Figure 163, right).

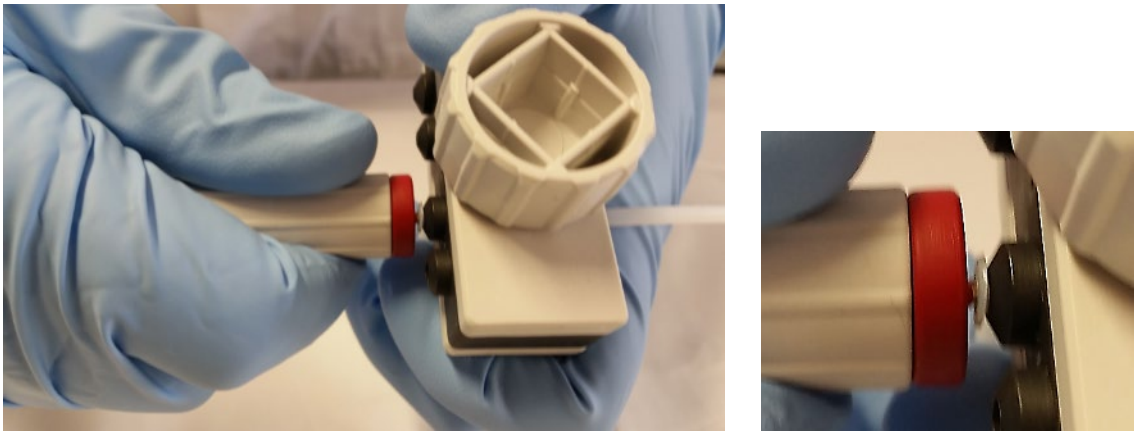


Figure 163: Flanging of the tubing.

The flange is made correctly, when the head has a mushroom shape and is uniform.
The figure below shows a flanging example.

Figure 164: Example for flanging.

Finally, a colour adapter can be put onto the NUT.

2) Preparing a flange connection with 1/16“ tubing

Materials:

NUT ¼"-28 (Part-Number: 10026)

Ring (Part-Number: 10438)

Flanging Tools (Easy-Flange-Kit, Part-Number: 10517)

Optional: Colour Adapter (Part-Number: 10028-10039)

Procedure:

First, cut the tube end square. Then connect NUT ¼"-28 and ring with the tube. Afterwards, insert the tube into the holder. This procedure is summarised in Figure 162.

Now, push the tube back in the direction of the holder using the hollow end of the flange pin in order to gain the correct projection required for the width of the flange. Then fix the tube in the holder.

Thereafter, put the 0.5 mm disc insert onto the flange pin and insert the pin into the tube hole (Figure 163, left). By rotating the flange pin and applying pressure in forward direction, the PTFE material is cold formed (Figure 163, right).

The flange is made correctly, when the head has a slight mushroom shape and is uniform; a flanging example is shown in Figure 164.

Optionally, a colour adapter can be put onto the NUT.

11. Troubleshooting/FAQ (Frequently Asked Questions)

This chapter gives you valuable information on questions and problems that might occur during the operation of your DEXTech Heat:

For the elimination of most errors, there are simple rules to follow:

- Most errors are caused by an obvious event.
- It is quite unlikely for two errors to occur at the same time.
- Errors can be found by systematically analysing the symptoms.
- Most errors have a defined starting point. Try to precisely determine the time when the error occurred first. Then the following questions are important: What happened before, during, and after the error occurred? The LCTech service engineers require this information in order to be able to locate the problem quickly.

ATTENTION:

- Never try to solve errors by unauthorized repair!
- Do not remove any covers or safety devices! After disassembling the system, reassemble the system to its original state.
- Checking and repairing electrical components of the system can only be carried out by LCTech staff. Any third-party repair attempt, which was not authorized by LCTech, voids any claims against LCTech and the device warranty!
- Contact:

LCTech GmbH
Daimlerstraße 4
84419 Obertaufkirchen
Germany
Fax: +49 8082 2717-100
E-mail: service@LCTech.de

11.1. Non-Reported Errors

Non-reported errors are not displayed by the system. This means there is no indication of an error in the software, consequently the system will not be stopped when such an error occurs. These errors can only be perceived visually by the user.

Non-reported errors include*:*

11.1.1 Sample Loop Runs Empty after Injection

Possible cause:

Leaks due to loose fittings or a torn off flange.

Troubleshooting:

- Check tubes on the injection valve.
- In case of a torn off flange: remove the remains from the valve seat and flange the tube again or fit a new collapsing ferrule and re-connect the tube.
  (Chapter 10.4 Replacement Tubes*).*

11.1.2 Column Not Tight Despite Tightened Column Tower

Indication: Wet column block or column is wet on the outside. This can be checked with the aid of a cloth.

Possible causes:

a) Missing seal in column 1 (Universal column) or in column 2 (Alumina column).

   Troubleshooting:

   - Check that seals are present in the column.
     ➔ If not, replace column and report to LCTech. 

b) Sealing cone of column is damaged.

   Troubleshooting:

   - Check that columns in the column tower are inserted in the correct order. If using the dummy column, columns 1 and 2 need to be inserted in the column tower with the bore showing at the front (see Figure 165).

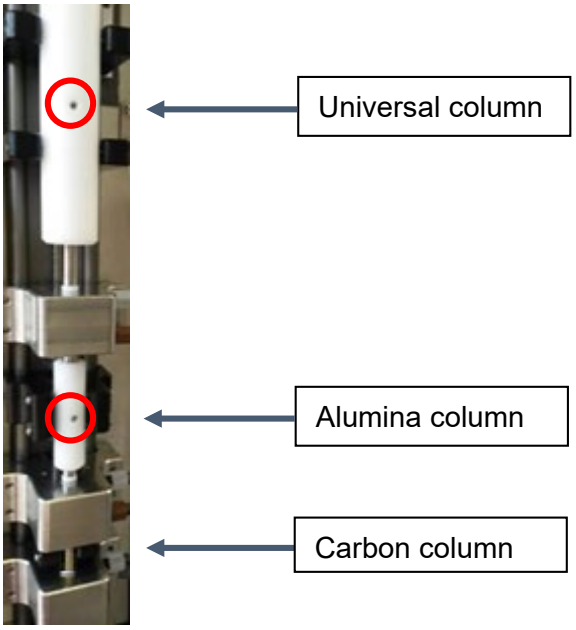


Figure 165: Column sequence and correct insertion of the dummy column. Columns 1 and 2 with the bore visible at the front.

- Check that the seat of the columns is plumb in the open (Figure 166, left) as well as in the closed (Figure 166, right) position.

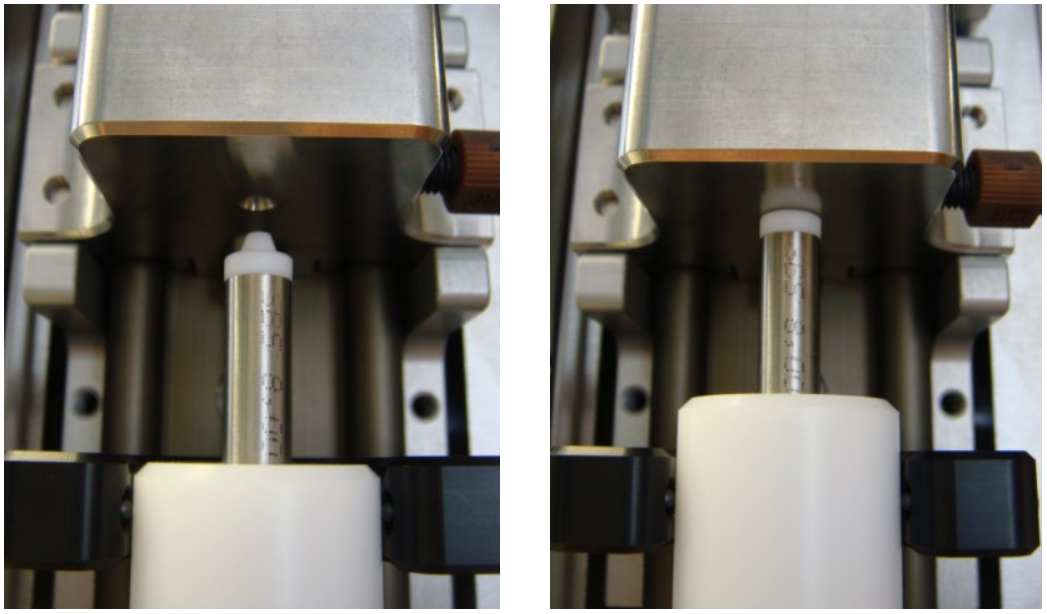


Figure 166: Checking the plumb position of the columns in the column tower. Left in the open position and right in the closed position.

In either case, please contact our LCTech service team.

11.1.3 Device Was Switched On, but Nothing Happens (Black Screen)

Possible cause: No power to the device.

Troubleshooting:

- Check the power supply (24 / 36 Volt DC), Figure 167.

Figure 167: Power supply DEXTech Heat.

- If no error can be found:

  → Please contact our LCTech service team.

11.1.4 Empty Solvent Supply Bottle

Possible cause: Solvent bottle not filled up, and consequently the solvent tubing has run empty.

Troubleshooting:

- Refill solvent supply bottle.
- Purge the respective solvent tubing (2-times) by using the appropriate software section (refer to *Chapter 5.7 Purge*).

NOTE: To ensure that the tubing is completely filled, the respective purging step must be executed twice.

11.1.5 The Column Tower Status Doesn't Change

Possible cause: The column tower has been closed without columns. In this case, the status of the button does not change from "*Open*" to "*Closed*".

Troubleshooting:

- Change the status of the button by pressing the "*Stop*" button in the software.
- Try to press the "*Open*" button again.
- If the column tower is open, put in the dummy columns and close the tower by pressing the "*Close*" button.

- If no error can be found:

  → Please contact our LCTech service team. 

Attention: If the system in not used for some time (for example 2 days, the weekend or for transport) the column tower should not be closed completely, to remove the pressure from the dummy columns and to prevent misalignment of the sensor. To do so close the column tower completely using the "close" button. Afterwards open the column tower so far that the dummy columns can be moved slightly with your hand.

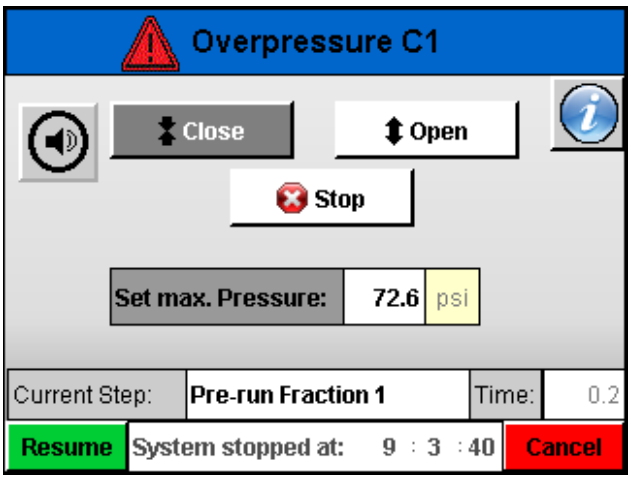
11.2. Reported Errors

Should any of the following errors occur, the system will stop immediately and an appropriate error message will be shown on the display.

List of reporting errors:

11.2.1 Overpressure Column 1

Cause: The following message will appear, if the set value for column 1 (Universal column, normally 5 bar or 72.6 psi) is exceeded in the processing steps: Conditioning 1, Pre-run C1, Pre-run Fraction 1 and Pre-run F2.



Error message 1: Overpressure of column 1 during the process.

Troubleshooting:

- Increase the value in the message window and continue processing.

ATTENTION: In case that the overpressure setting for column 1 is increased observe all the process steps in which column 1 is included during the run. Due to the increase in value, the pressure can rise rapidly causing column 1 to leak or a tube to spall thereby creating a leakage. For safety reasons, the following warning will appear in the error window.

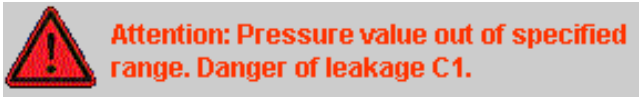
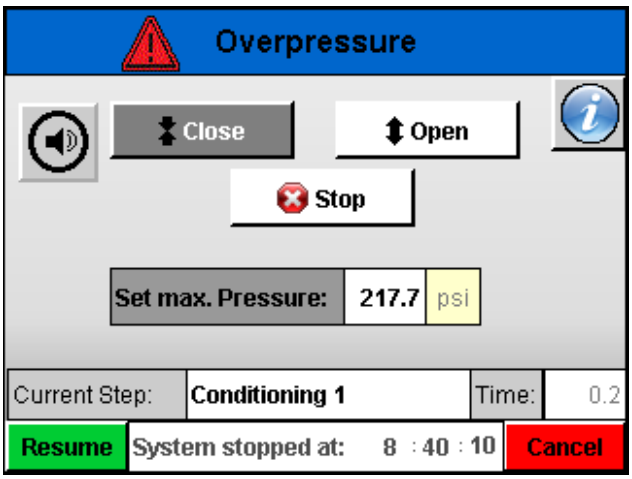


Figure 168: Safety warning when the pressure limit of column 1 is increased.

- If the value has to be raised to the maximum pressure of 15 bar (217.7 psi), please refer to the following *section 11.2.2* Overpressure .

11.2.2 Overpressure

General: This message (see Error message 2) appears when the system exceeds the set standard maximum pressure value, which is normally set at 15 bar or 217.7 psi.



Error message 2: Overpressure fault in the system.

Possible Causes:

- For the previous error message for column 1: Column 1 (Universal column) is overloaded and, as a result, the tube is blocked.
  Troubleshooting:
  1. Reduce the sample load.
  2. Locate the blocked tube using the tubing diagram (see *Chapter 9* Fluidics) or press the „*I*“ button (Error message 2, left) and remedy.

- Squashed or bent tubes.
  Troubleshooting:
  - Renew tubes *(Chapter 10.4 Replacement Tubes*).

- The maximum pressure is set too low.
  Troubleshooting:
  - Increase maximum pressure (15 bar/ 217.7 psi).

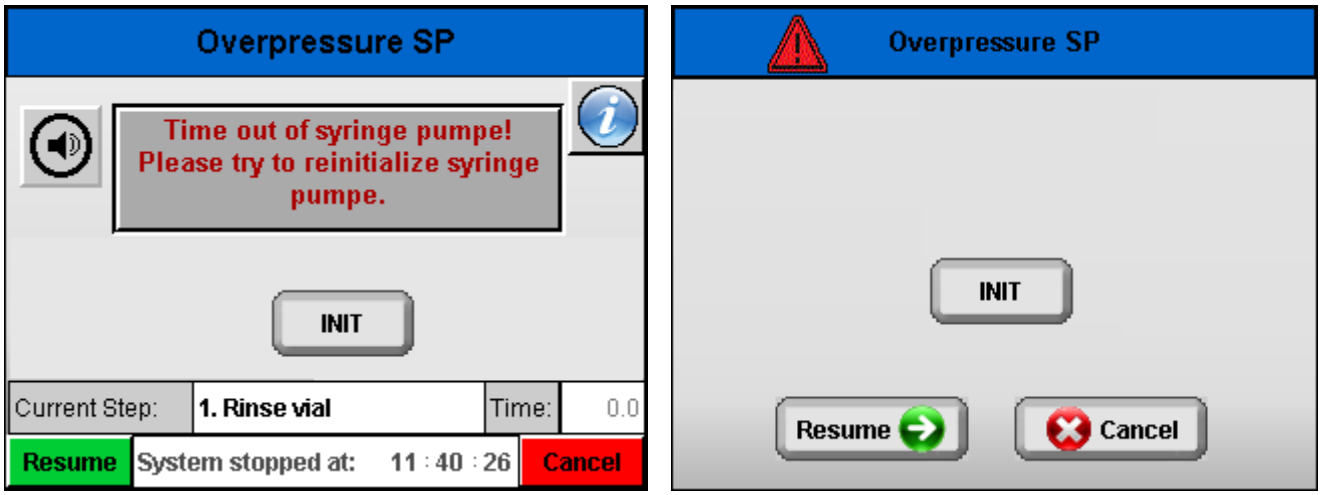
- Incorrect valve position.

  Procedure:

  - Identify in the internal area of the software (*“Internal“* → *“Valve“*, see *Chapter 10.3.1 Internal Area point i*) the respective valve via manual switching of the valves.
  - Then contact the LCTech service team.

11.2.3 Overpressure Syringe Pump

General: Error message 3 appears if the syringe pump incurs overpressure.



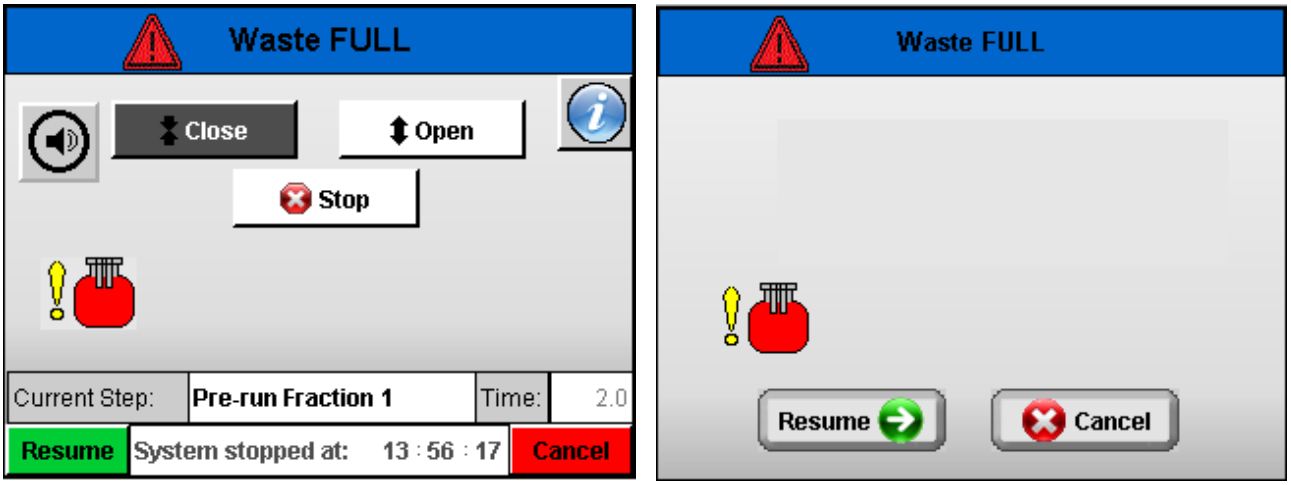
Error message 3: Error page for syringe pump.

Troubleshooting:

- 1. Continue process ("*Resume*" button).
- 2. If error message is displayed again:
  - During the process: Press "*INIT*" button. Afterwards, the syringe should be reinitializing.
  - Outside of the process: Abort current step („*Cancel*") and go to the internal section (point h) syringe pump). Then execute the function control of syringe pump by pressing the "*INIT*" button (Figure 145).
- 3. Alternative
  - Control possible: Repeat previous step.
  - No control possible:
    Contact the LCTech service team.

11.2.4 Waste Full

Cause: The following error message appears only if the system is equipped with the optional float switch for the solvent waste and the sensor has detected a signal at the disposal flask.



Error message 4: Error message "Waste full". Left: during the process and right: outside of sample preparation.

Troubleshooting:

A) When using a liquid level sensor for the waste:

Check whether:

1. Waste bottle is full.
   → If yes, empty waste bottle.

Disposal
Please observe local regulations for collection and disposal of laboratory waste

2. Sensor not plugged in.
   → Connect plug.

3. Float body is stuck (blocked by waste container or waste tube) and has erroneously caused a signal. The float should be slightly springy and, at rest, have a distance of around 6 - 7 mm to the collar of the guide bar, as shown in the figure below.

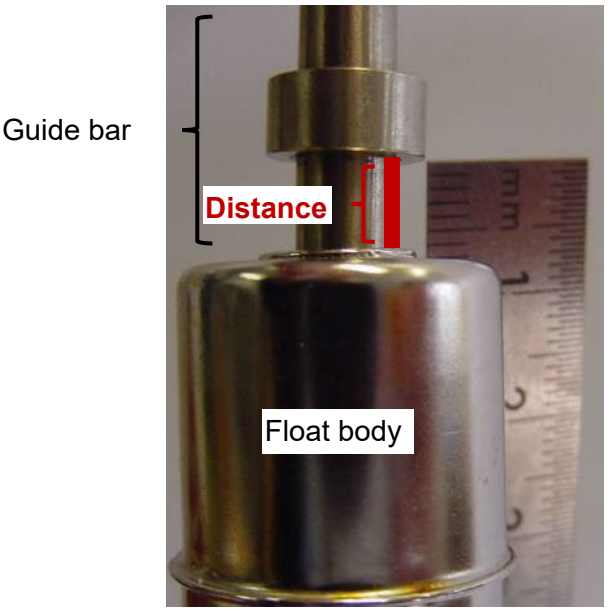


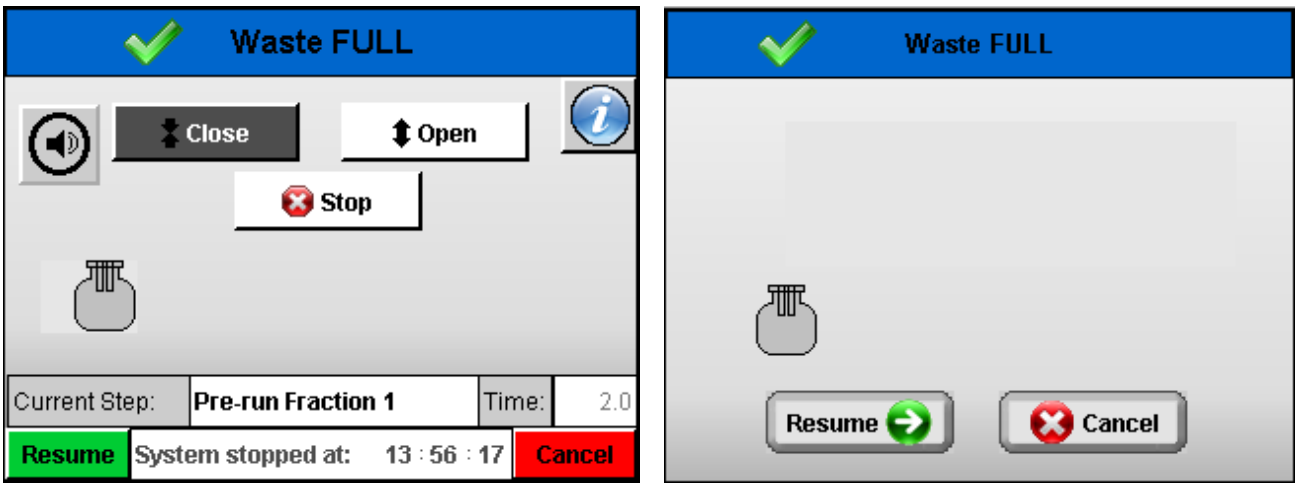
Figure 169: Distance of float body to collar of guide bar.

→ free up and down movement of float body.

B) If you do not use a level sensor:

A so-called "dummy plug" needs to be inserted in place of the level sensor at the sensor's usual position. This dummy plug is part of the standard equipment.

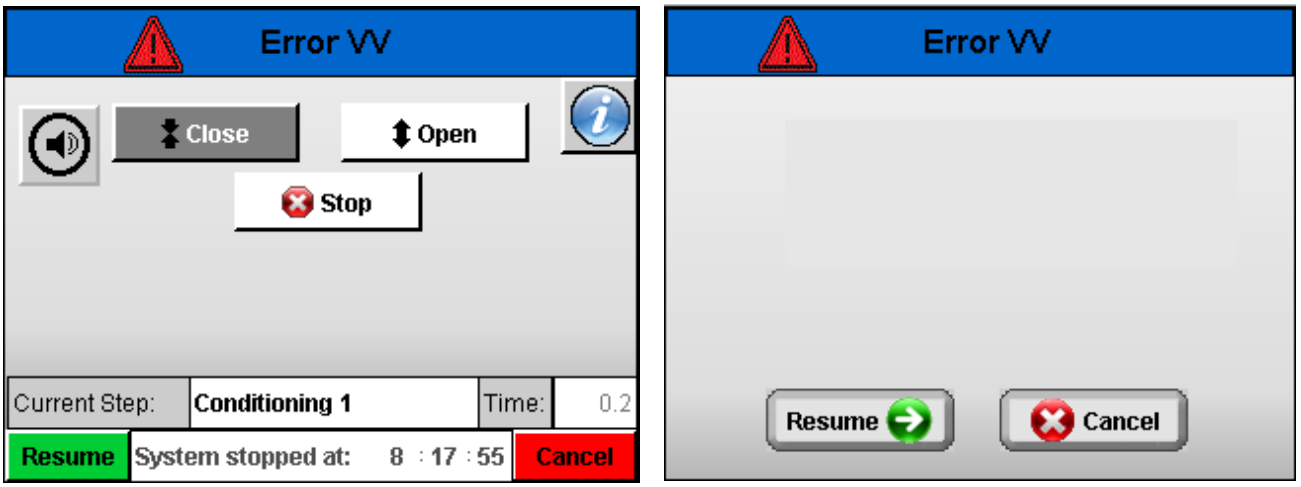
NOTE: The fault is corrected, when a green check mark or an empty waste container is displayed in the software, see Error message 5.



Error message 5: Corrected fault "Waste full".

11.2.5 Error IV, VV, LsmV, ZV1, ZV2

Cause: Error message 6 appears when a valve position cannot be approached correctly.



Error message 6: Reporting a positioning fault and indicating the respective, affected valve. Left during the process and right outside of the sample preparation.

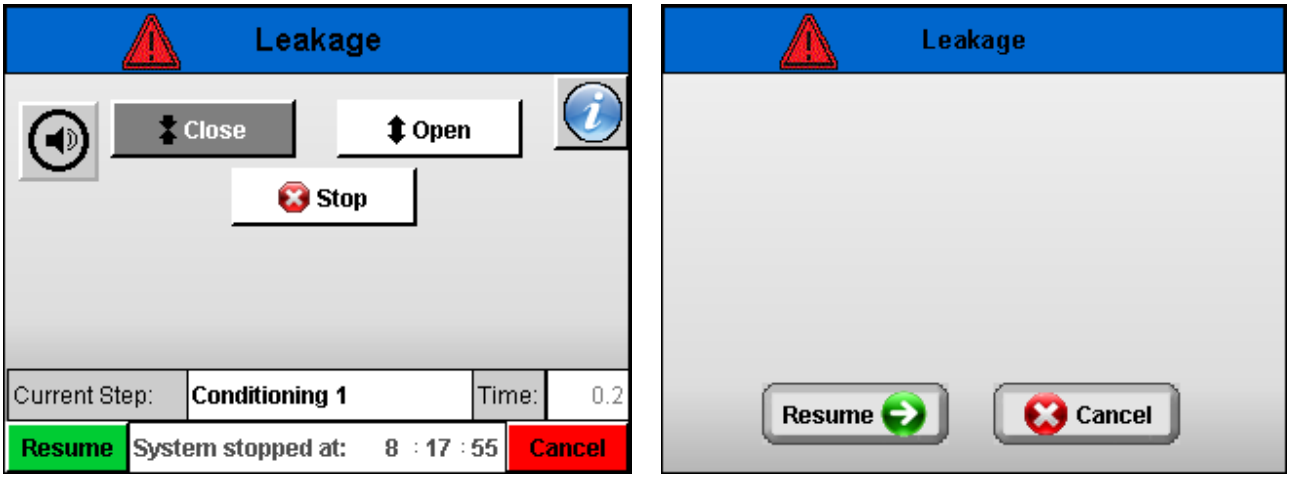
Troubleshooting:

- In the internal area ("*Internal*" → "*Valve*", see *Chapter 10.3.1 Internal Area point j*) press the "*Reset*" button and switch the valve manually to various positions:
  - IV: Injection valve (Inj.Valve, maximum 5 positions)
  - VV: Distribution valve (V. Valve, maximum 6 positions)
  - LsmV: Solvent valve (Lsm. Valve, maximum 6 positions)
  - ZV1: Central valve 1 (Z. Valve 1, maximum 5 positions)
  - ZV2: Central valve 2 (Z. Valve 2, maximum 5 positions)

➔ If the error re-occurs, please contact the LCTech service team.

11.2.6 Leakage

Cause: This message (Error message 7) is triggered by a leak in the system. The sensor detects solvent in the drip tray.

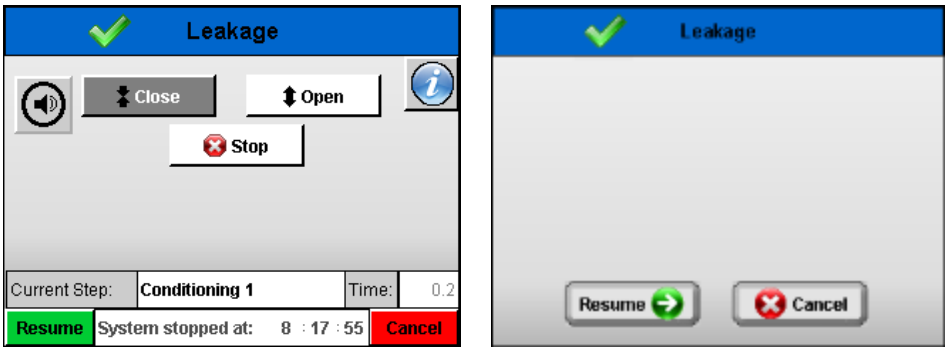


Error message 7: Display of a leakage fault. Left: error during processing and right outside of processing.

Troubleshooting:

- Determine cause and location of the leak see *Chapter 9* Fluidics or press „*I*“ button, see Error message 7 left) and remedy.
- In the event of a torn off flange, remove the remains from the valve seat and flange the tube again or fit a new collapsing ferrule and re-connect the tube (*Chapter 10.4 Replacement Tubes*).
- If solvent escapes from the overflow of a valve head, please contact the LCTech service team.
- If the columns leak, the seat of the columns needs to be checked (see *Chapter 11.1.2 Column Not Tight Despite Tightened Column Tower*).
- After resolving the leak, the drip tray must be emptied (*Chapter 10.1: Emptying the Drip Tray*).

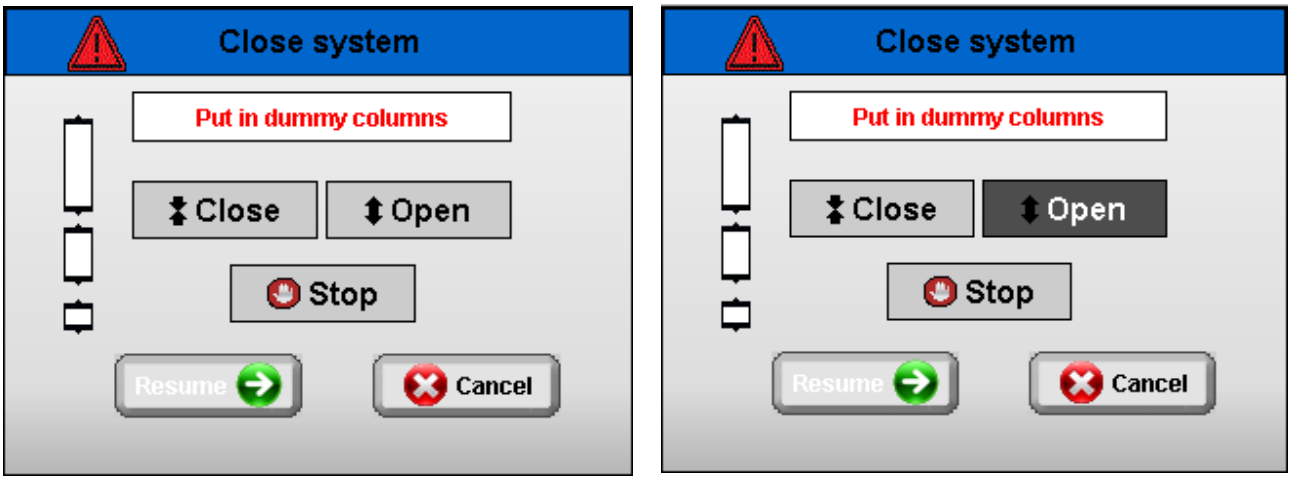
NOTE: The fault is corrected, when a green check mark is displayed in the software, see following resolved error message.



Error message 8: Resolved leakage fault indicated by display of the green check mark.

11.2.7 Lock Column Tower

Cause: Error message 9 appears when the column tower is not completely locked.



Error message 9: Displayed when column tower is open.

Troubleshooting:

- Lock column tower, if necessary open slightly first and then lock again properly.

Caution: Never reach directly into moving machine parts or into gaps, which are provided for mechanical movements.

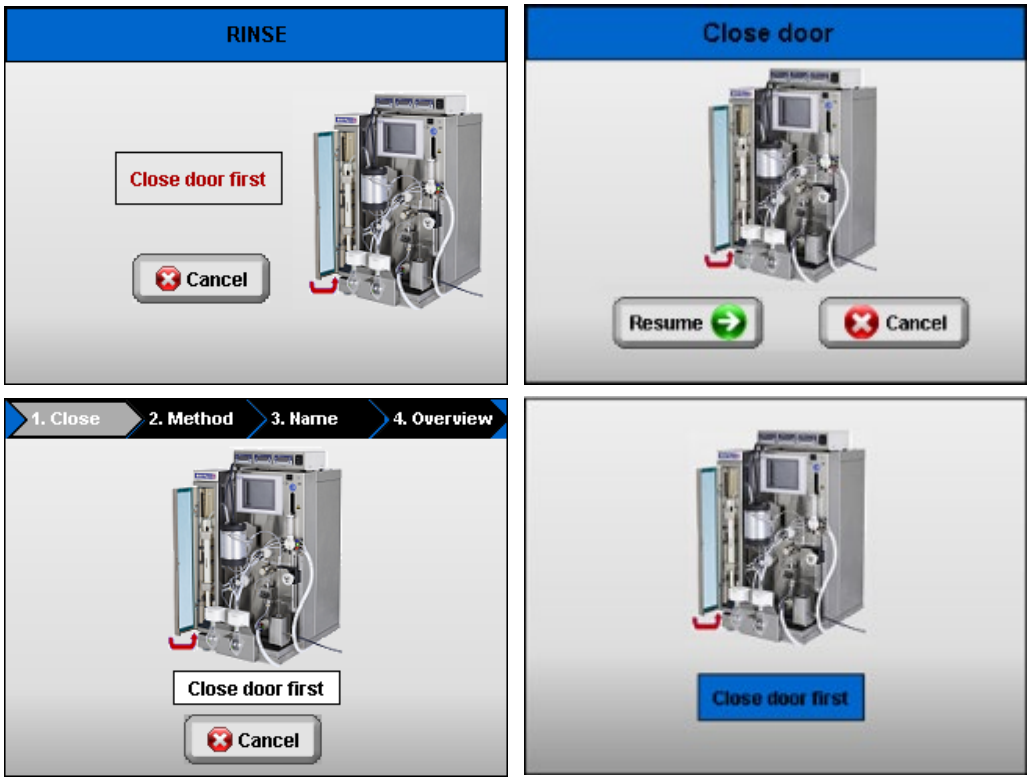
- If the column tower is properly locked, but the fault persists, then the positioning sensor has been overridden.
  → Please contact the LCTech service team.

Attention: If the system in not used for some time (for example 2 days, the weekend or for transport) the column tower should not be closed completely, to remove the pressure from the dummy columns and to prevent misalignment of the sensor. To do so close the column tower completely using the “Close“ button. Afterwards open the column tower again until the dummy columns can be moved slightly with your hand.

NOTE: In order to lock the column tower, the front door must be closed. Otherwise a prompt will appear with the request to close the front door (see Error message 10).

11.2.8 Close Door

General cause: The door contact switch, which is located behind the upper hinges of the front door, does not receive the “Closed” signal. This is indicated in the following error message.



Error message 10: Prompt to close the front door of the device

This error message is displayed when changing columns and serves as a safety feature to ensure that the front door of the unit is closed during opening / locking of the column tower.

To continue, you need to close the front door of the appliance.

Caution: Never reach directly into moving machine parts or into gaps, which are provided for mechanical movements.

Possible triggers:

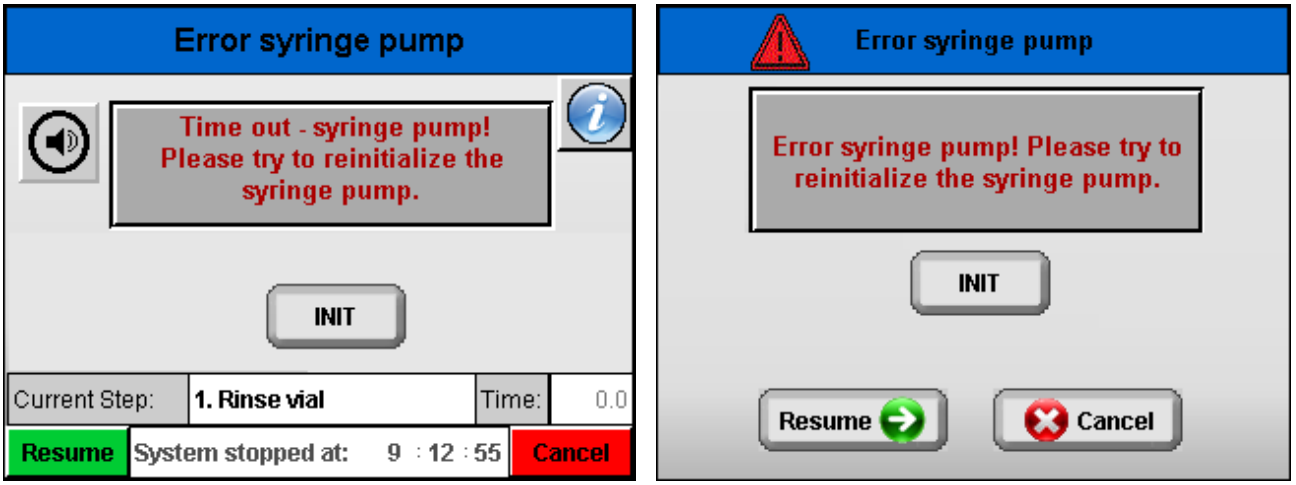
- Front door not closed.

Troubleshooting:

- Close the front door.
- The door locking sensor has failed.
  → Please contact the LCTech service team.

11.2.9 Error Syringe Pump

General information: This error message appears on the display if the syringe pump has not reacted to a signal for a certain period of time.
Error: Time out - syringe pump. Please try to re-initialize the syringe pump.



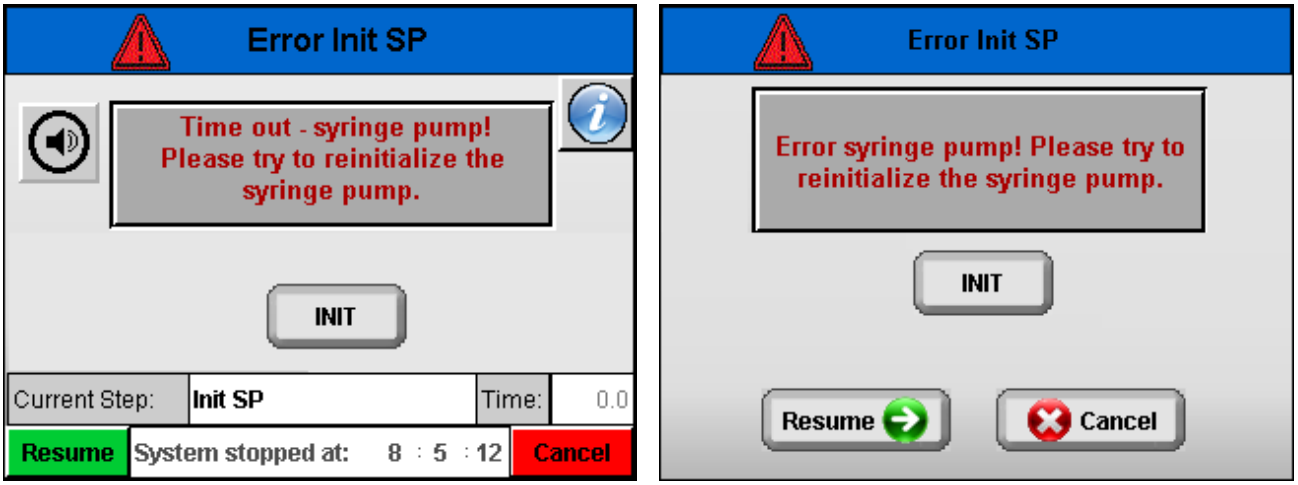
Error message 11: Syringe pump error. Left: during the process. Right: outside of the clean-up process.

Troubleshooting:

- 1. Continue process ("*Resume*" button).
- 2. If error message is displayed again:
  - During the process: Press "*INIT*" button. Afterwards, the syringe should be reinitializing.
  - Outside of the process: Abort current step ("*Cancel*") and go to internal section (point h) syringe pump). Then execute the function control of the syringe pump by pressing the "*INIT*" button (Figure 145).
- 3. Alternative
  - Control possible: Repeated previous step.
  - No control possible:
    → Please contact the LCTech service team.

11.2.10 Error Init SP

General information: The Error SP Init will appear (Error message 12) if the syringe pump loses its initial value during the run.



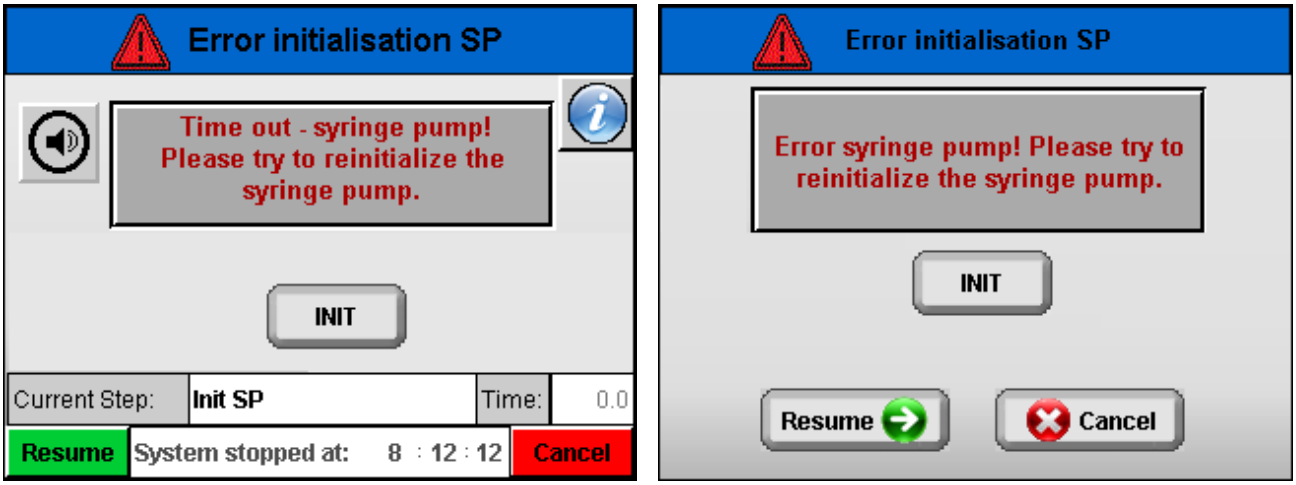
Error message 12: Error Init SP. During (left) and outside (right) of the process.

Troubleshooting:

- 1. Error message:
  - During the process: Press "*INIT*" button. Afterwards, the syringe should be reinitializing.
  - Outside of the process: Abort current step ("*Cancel*") and go to internal section (point h) syringe pump). Then execute the function control of the syringe pump by pressing the "*INIT*" button (Figure 145).
- 2. Alternative
  - Control possible: Repeat previous step.
  - No control possible:
    → Please contact the LCTech service team.

11.2.11 Error Initialization SP

General information: The error „Error initialization SP" (Error message 13) will appear, if the syringe pump was not initialized during the boot process.



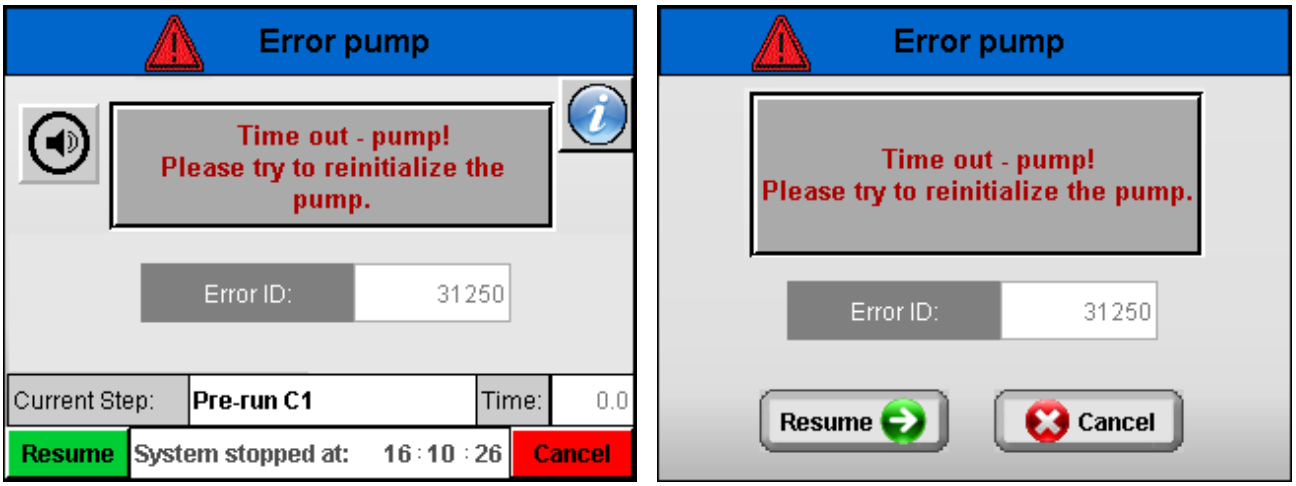
Error message 13: Initialization error of the syringe pump. During (left) and outside (right) of the clean up process.

Troubleshooting:

- 1. Error message:
  - During the process: Press "*INIT*" button. Afterwards, the syringe should be reinitializing.
  - Outside of the process: Abort current step ("*Cancel*") and go to internal section (point h) syringe pump). Then execute the function control of the syringe pump by pressing the "*INIT*" button (Figure 145).
- 2. Alternative
  - Control possible: Repeat previous step.
  - No control possible:
    → Please contact the LCTech service team.

11.2.12 Error Pump

General information: The error will appear (see Error message 14) if the piston pump does not react to an internal signal. The error is triggered by an internal pump error.



Error message 14: Error of the piston pump. During (left) and outside (right) of the clean up process.

Troubleshooting:

- 1. Error message:
  - Abort current step ("*Cancel*") and go to internal section (point j pump) try to control the GPC-pump manual.
  - Switch device off and then on again.
- 2. Alternative
  - Control possible: Repeat previous step.
  - No control possible:
    → Please contact the LCTech service team.

11.2.13 Power Failure

General information: There are several ways to proceed after a power failure happened during the clean-up process.

If a power failure happens during the transfer of the sample to the sample loop or during the rinsing of the sample vial.

1) Back transfer of the sample: In the rinse window, an additional button for the back transfer of the sample to the sample vial will appear (see Figure 170).

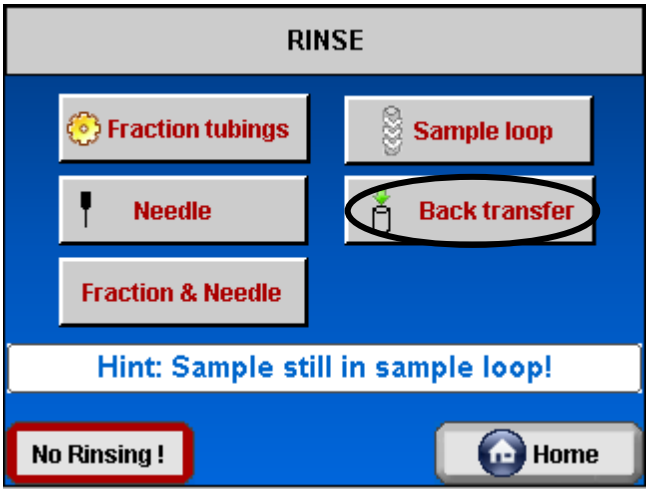


Figure 170: Sample "back transfer" button in the rinse window.

Tip: The sample back transfer is only recommended if the sample transfer to the sample loop has only just started.

2) Rinse the system and start a new sample. With this option the last sample will be lost.

INFORMATION: The decision as to which option to use is at the discretion of the user.

Option 1) Back transfer of the sample

After pressing the “B*ack transfer*” button, the following window will appear in the display.

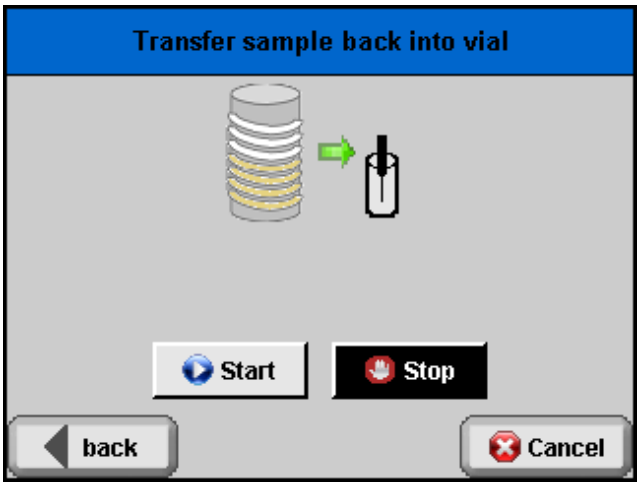


Figure 171: Back transfer display.

The back transfer process consists of the following steps:

1) The syringe pump draws a defined volume of n-Hexane.
   The volume depends on the step at which the clean-up process was interrupted. For example, if the clean-up process was interrupted while the syringe has drawn the first rinsing solution of the sample vial:
   Volume = Sample volume + volume of the first rinsing solution
2) Afterwards, the sample is transferred back into the sample vial.

As long as this process is running, the “*back*”- and “*Cancel*” buttons appear in black and are inactive. The process can be interrupted with the “*Stop*” button (Figure 173) at any time.

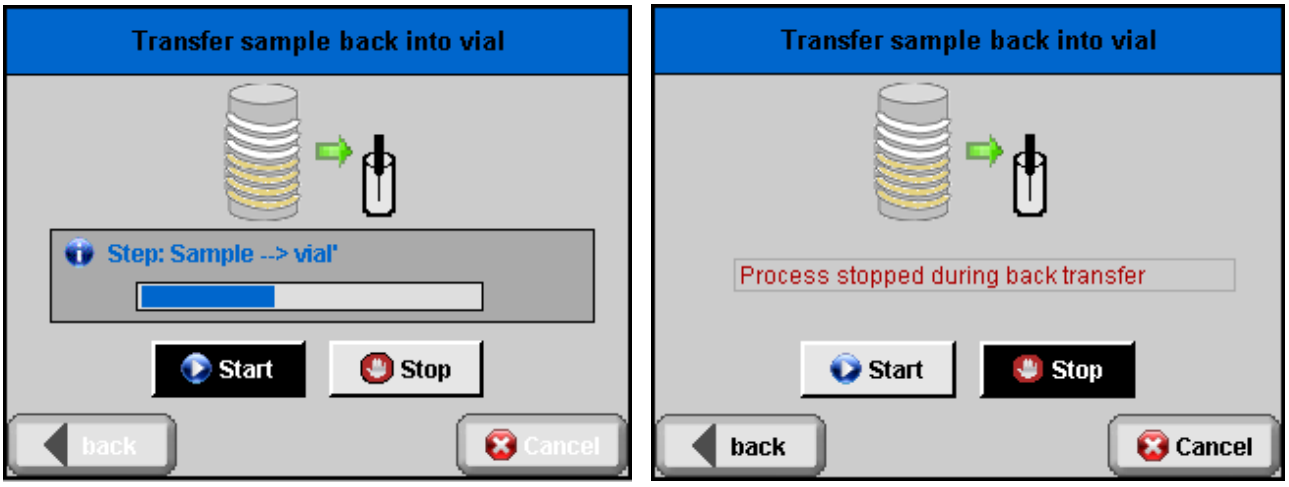
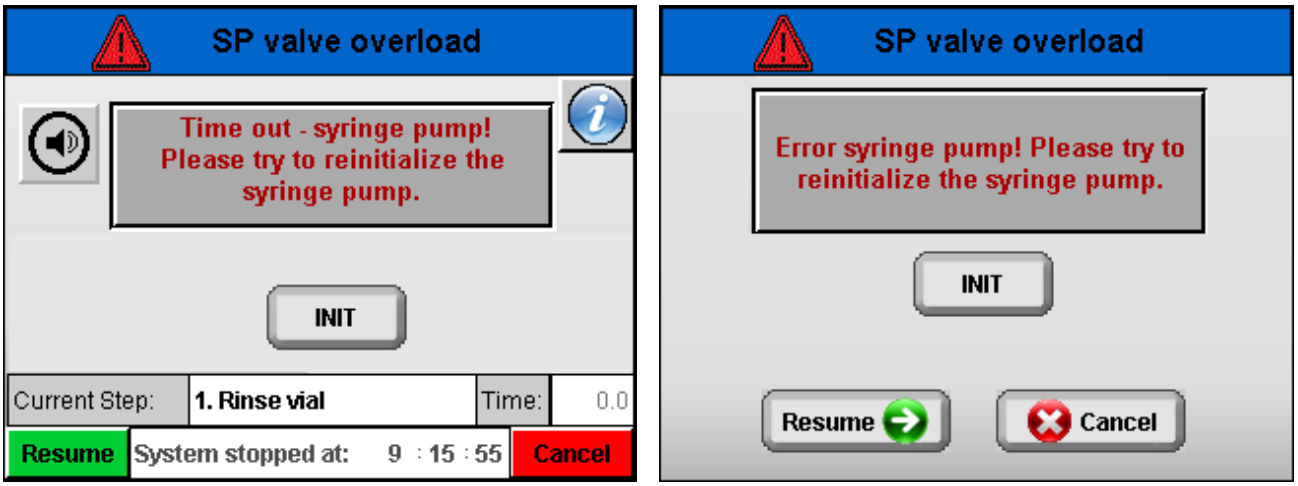


Figure 172: Back transfer of the sample. Left active process and right during interruption.

At the end of the process, the sample can be used a second time.

11.2.14 Error SP Valve Overload

General information: Error message 15 appears if the syringe pump valve overloads during the run with the effect that the valve does not turn afterwards.



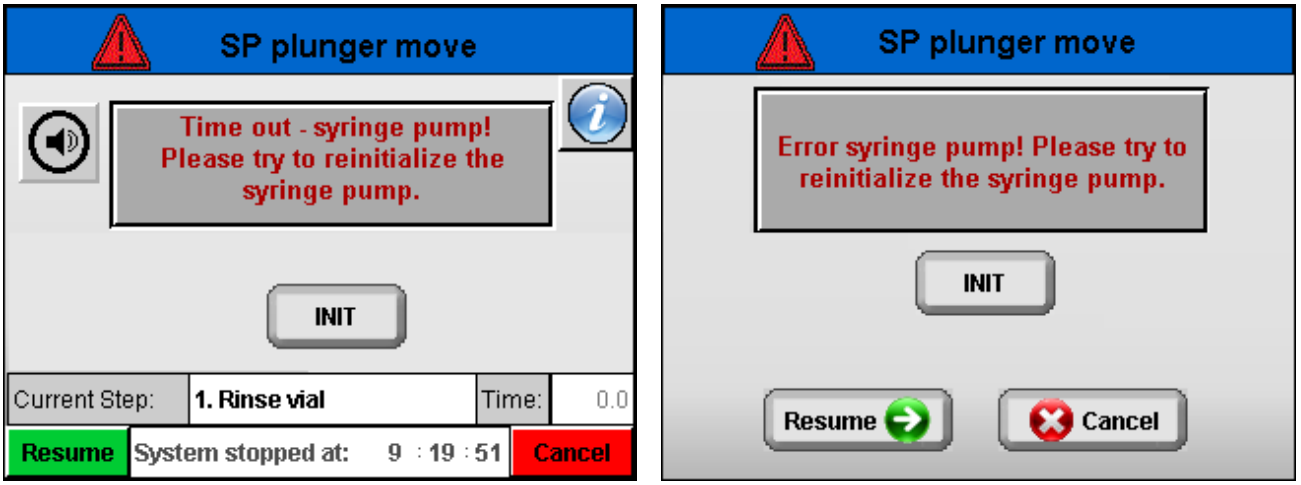
Error message 15: Error SP valve overload. During (left) and outside (right) of the process.

Troubleshooting:

- 1. Error message:
  - During the process: Press "*INIT*" button. Afterwards, the syringe should be reinitializing.
  - Outside of the process: Abort current step ("*Cancel*") and go to internal section (point h) syringe pump). Then execute the function control of the syringe pump by pressing the "*INIT*" button (Figure 145).
- 2. Alternative
  - Control possible: Repeat previous step.
  - No control possible:
    → Please contact the LCTech service team.

11.2.15 Error SP Plunger Move

General information: The Error SP plunger move will appear (Error message 16) if the syringe pump plunger do not move during the run.



Error message 16: Error Init SP. During (left) and outside (right) of the process.

Troubleshooting:

- 1. Error message:
  - During the process: Press "*INIT*" button. Afterwards, the syringe should be reinitializing.
  - Outside of the process: Abort current step ("*Cancel*") and go to internal section (point h) syringe pump). Then execute the function control of the syringe pump by pressing the "*INIT*" button (Figure 145).
- 2. Alternative
  - Control possible: Repeat previous step.
  - No control possible:
    → Please contact the LCTech service team.

12. Reshipment of the Device

If you need to return your LCTech device please contact the LCTech Service team first. 

13. Service Parts

In case parts need to be replaced, please use LCTech original spare parts only. All parts have been carefully selected and tested and ensure proper function.

Below you will find the part to be serviced.

Part-Number	Description
15324 includes:	Maintenance Kit for DEXTech Heat
2 x 10212	Primary seal, preparative
2 x 10213	Secondary seal, preparative
2 x 10214	Centre ring, preparative
3 x 12971	Rotor seal 10-Port-M-Pos
2 x 15331	Rotor seal 6-Port-M-Pos Dead-End Path
14663	Grease

14. Parts and Accessories

In case parts need to be replaced, use LCTech original parts only. All parts have been carefully selected and tested and ensure proper function.

Below you will find the part to be serviced.

14.1. Spare Parts

Pump

Part-Number	Description
10212	Primary seal, preparative
10213	Secondary seal, preparative
10214	Centre ring, preparative
10215	Ceramic plunger for pump, preparative
10216	Check valve
11421	Timing belt 2.5-200
14663	Grease

Upgrade Check Valve Cartridge, double ball 1/8”

Part-Number	Description
16592	Check Valve Cartridge, double ball 1/8”
16593	Valve housing, IN
16594	Valve housing, OUT

Tubes 1/8“

Part-Number	Description
10042	PTFE Tube ⅛“, 1 m
10027	Nut, PPS, 1/8",1/4"-28
10437	Ring, PP, flanged 1/8"

Tubes 1/16“

Part-Number	Description
10043	PTFE Tube 1/16“, 1 m
10026	Nut, PPS, 1/16",1/4"-28
10438	Ring, PP, flanged 1/16"

Heated Parts

Part-Number	Description
16626	PFA Tube 1/8", Heated, Needle to Valve & Valve to 1st Column, inclusive Heaters, Cable and Plug
16522	Sample Holder, Heated
16498	Sample Loop 20mL, Heated

Valve

Part-Number	Description
12548	Valve 10-Port-M-Pos
12971	Rotor seal 10-Port-M-Pos
14493	Stator 10-Port-M-Pos
13643	Valve 6-Port-M-Pos Dead-End Path
15331	Rotor seal 6-Port-M-Pos Dead-End Path
15330	Stator 6-Port-M-Pos Dead-End Path

Electronic accessories

Part-Number	Description
11556	Battery for PLC
12283	G-Fuse, flink 4A
14316	Flink Fuse 1A

General accessories

Part-Number	Description
10020	Solvent filter 10 µm sinter filter
12287	Dummy for liquid level switch
13381	Safety exhaust filter
13762	Protection film for power panel
14262	Adapter for Dummy-SMART
15634	Needle double-walled, DEXTech Heat
16819	Report function for DEXTech Heat

Kits

Part-Number	Description
16039	First Aid Kit (assorted collection of nuts, ferrules, tubings and fittings) Easy Flange Kit P/N 10517 is highly recommended
10517	Easy Flange Kit
12539	Tool-Kit

14.2. Optional Parts

Racks

Part-Number	Description
14951	Fraction Rack for two rotary evaporator flasks NS 14/23
15542	Fraction Rack for two rotary evaporator flasks NS 29/32
16042	Rack for up to four solvent bottles (max. diameter 103 mm/usually 1 L flasks)

Liquid Level Switch

Part-Number	Description
11587	Liquid Level Switch

14.3. System

Part-Number	Description
16800	DEXTech Heat

14.4. Column

Part-Number	Description
19513	SMART column (25 pcs/pkg) (up to 1.5 g fat) requires: 14262 Adapter for dummy-SMART
19512	Standard column (25 pcs/pkg) (up to 5g fat)
19511	Universal column Glas (25 pcs/pkg) (up to 5g fat)
20085	Universal column plastic (100 pcs/pkg) (up to 5g fat)
15433	Alumina column glass (25 pcs/pkg)
20087	Alumina column plastic (100 pcs/pkg)
15242	Carbon column (10 pcs/pkg)

14.5. Dummy Column

Part-Number	Description
13708	Dummy column Pos 1
14438	Dummy column Pos 1 (SMART) require: 14262 Adapter for dummy-SMART
13711	Dummy column Pos 2
13713	Dummy column Pos 3

15. Version History

Version-No	Date	Description	SW Version
1.1	7.05.2019	Update: New set-up Dioxin only	2.0
1.2	09.10.2019	Update: 13. Parts and Accessories	2.0
1.3	06.12.2019	Updated: Service interval	2.0
1.4	06.05.2022	New Functions: Quickstart, Combine and Fraction 1-C1	2.1