

From Eye to Insight



EM CPD300

Automated Critical Point Dryer

Instructions for use

Leica Microsystems CMS GmbH; Instructions for use 167180032, Revision 2018-12



Important Note

Leica Microsystems CMS GmbH reserves the right to change technical specifications as well as manufacturing processes without prior notice. Only in this way is it possible to continuously improve the technology and manufacturing techniques used to provide our customers with excellent products.

Any copyrights of this document are retained by Leica Microsystems CMS GmbH. Any reproduction of text and illustrations (or any parts thereof) by printing, photocopying, or other methods (including electronic systems and media) requires express prior permission in writing.

Issued by:

Leica Microsystems CMS GmbH
Ernst-Leitz-Strasse 17-37
D-35578 Wetzlar, Germany
www.leica-microsystems.com

Leica EM CPD300

Operating Manual

Leica EM CPD300 Serial Number:

Date of purchase:

For the instrument serial number, please refer to the name type label on the back of the instrument!



**Please read this instruction manual carefully before operating the instrument.
For Research use only!**

Foreword

This technical documentation is intended to provide essential information about the proper operation and servicing of the EM CPD300 critical point dryer.

Service and operating staff must familiarize themselves with all components of the system before commissioning. Particular attention must be paid to the aspect of safety.

Please retain this operating manual for future reference.

Texts, schedules and tables may not be copied, reproduced, or divulged to third parties without the express consent of Leica Microsystems CMS GmbH.

In addition, all generally applicable legal and otherwise binding regulations for preventing accidents and protecting the environment must be observed by the user and communicated to all users.

Table of contents

1. Introduction	3
2. Safety	4
2.1 Safety concept of Leica Microsystems CMS GmbH	4
3. Installation and Warranty	6
3.1 Installation	6
3.2 Warranty	8
4. Method	9
4.1 Critical Point Drying Method	9
5. Description and Specification	11
5.1 Technical specifications	11
6. Preparation of Specimens	12
7. Automatic Operation of EM CPD300	13
7.1 Screen descriptions	13
7.2 Programming by example	18
7.3 Programming the EM CPD300 procedure	27
7.4 Filling the Sample Chamber with exchange fluid	27
7.5 Inserting Specimen into Sample Container	27
7.6 Preset of Cooling Temperature of the Sample Chamber	28
7.6.1 CO ₂ -Bottle Temperature / Pressure Function	28
7.7 Starting the EM CPD300 auto procedure	29
7.8 Time and timer function	29
8. Manual Operation of EM CPD300	34
8.1 Screen descriptions	34
8.2 Filling the chamber with exchange fluid	36
8.3 Inserting of Specimen into Sample Chamber	36
8.4 Cooling of the Sample Chamber	36
8.5 Filling the CO ₂ into Sample Chamber	37
8.6 Mixing the two liquid media	37
8.7 Exchange media mixture from Sample Chamber	37
8.8 Heating the Sample Chamber	38
8.9 Releasing gaseous CO ₂ from Sample Chamber	38
9. Removing Dried Specimen	38
10. Switching off the Unit	38
11. Setting Panel	39
12. CO₂-Bottle Temperature / Pressure Function	40
13. Adjustments of Pressure Threshold for Bottle Empty Function	41
14. Software Update	43
15. Service Notes	45
15.1 Gas-Out filter maintenance	45
15.2 General maintenance	46
16. User Maintenance	47
16.1 Poral filter replacement	47
16.2 Cleaning	49
17. Warnings and Troubleshooting	50

1. Introduction

In order to ensure the safety of service technicians and operators, and to prevent any damage to the EM CPD300 critical point dryer, it is essential to carefully read this manual before beginning any work with the system.

This Operating Manual is intended to help the user understand the system more completely, to use it within the specified limits of its working capabilities, and to maintain in accordance with its physical parameters.

This user manual includes important information regarding proper installation, operation, troubleshooting and repair. Following these instructions will help to prevent hazards, reduce repair and downtime costs, and prolong the system's service life.

Symbols in this manual and their meaning:



Danger!

All paragraphs in the Technical Documentation that contain instructions regarding possible hazards are identified with this symbol.

Non-observance these alerts may result in serious injury! Users of the instrument must comply with instructions at all times.



Caution!

This symbol alerts the user to important information which may endanger staff or result in damage to the system if it is ignored.



Note!

This symbol indicates further information relating to a previous explanation, which does not have a safety-critical function. However, it is important to follow this information to ensure that the system functions optimally.

To download the EC Declaration of Conformity, use this link

<https://www.leica-microsystems.com/products/sample-preparation-for-electron-microscopy/p/leica-em-cpd300/downloads/>

If you have any further questions or require technical support, please contact your country's Leica branch office or your local contact person directly. The appropriate contacts can be found on the Internet under:

<http://www.leica-microsystems.com>

2. Safety



Warning!

The EM CPD300 critical point dryer can be operated simply and safely, if the system is operated according to the operating manual.

2.1 Safety concept of Leica Microsystems CMS GmbH

The use in accordance with regulations of the EM CPD300 critical point dryer consists of critical point drying of biological or industrial samples with minimal deformation of the specimens for subsequent SEM analysis.

Any other application without written approval of the manufacturer can lead to damages and injuries of instrument and user. The manufacturer refuses to take any responsibility for damage caused thereby.

The sample pressure chamber with a maximum working pressure of 150bar is secured with a Software and Hardware Cut Off function.

Hardware controlled cut off

Bursting membrane at 105bar at 20 °C (+/-10%)

Software controlled cut off

Pressure at 80bar and temperature at 45°C

For safe operation it is absolutely necessary to set up and connect the device according to the instructions in Chapter 3.

General safety regulations

Generally, the following safety regulations apply to the handling of the EM CPD300 critical point dryer:

- Every user is responsible for her / his own health.
- Only instructed users, authorized by the customer are allowed to work at or with the system.
- For all interaction with the EM CPD300 the user must wear protective clothing prescribed in the respective environment.
- It is strictly prohibited to alter or remove protective equipment or covers of any kind.
- Every user must be trained in safe handling of the process gases and fluids that are utilized in the EM CPD300 unit.
- After every repair the user (technician) must verify a flawless state of the system by a test run.
- Leica can guarantee full operation of the system only if original spare parts according to the part lists are used.
- Careful operation and preventive maintenance of the EM CPD300 reduces maintenance costs and assures reliable operation.
- By means of frequent checks and prompt rectification of even small damages, considerable damage can often be avoided.
- Observation of this operating manual and all notes installed on the system serves your own safety.

3. Installation and Warranty

3.1 Installation

Required space for setting up the unit: width 340 mm, depth 583 mm.

Operating room temperature: 20 – 35C° at 1bar (CO₂ bottle limit)

Relative humidity: 5 – 90%

To allow adequate intake of cooling air required for the refrigeration unit and due to the inflexibility of the high-pressure gas hose, a free space of 150 mm has to be provided on the backside of the instrument.

Connection of CO₂ Container

It is absolutely **necessary** to use a pressurized gas container with a pipe (feed pipe), so that the medium (CO₂) can reach the specimen pressure chamber in liquid form. It is important here to ensure that the CO₂-Container is not equipped with a residual pressure valve.

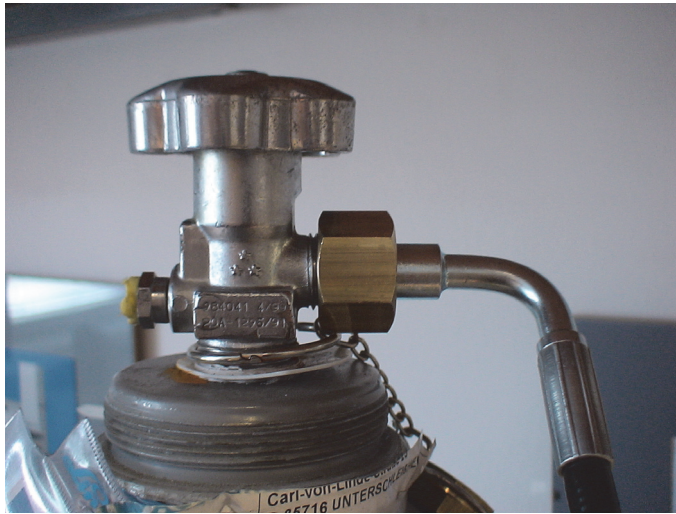


In order to prevent accidents, the CO₂-pressurized gas container must be securely fastened in an upright position.

Screw the high-pressure gas hose onto the CO₂ IN connection.



Connect the other end of the high-pressure gas hose to the gas pressure container.



The high-pressure gas hose and seal are included in the accessory set of the unit.

Electrical Connection

Check assigned connecting voltage of the unit.



Connect power cable to wall socket.

3.2 Warranty

The EM CPD300 is covered by a WARRANTY in accordance with the conditions of sale. If functional errors should occur or if the components of the system sustain damage that is subject to warranty coverage during the warranty period, the manufacturer will repair or replace the faulty components following examination thereof.

The manufacturer warrants for the system in its original configuration.

Only original replacement parts may be used. The manufacturer accepts no liability for damage caused by use of other replacement parts.

Caution!



The environmental conditions that were agreed contractually and determined at the time of installation must be maintained.

The manufacturer will not accept liability for damage caused by misuse of the system or its use for purposes other than the intended use, nor for damage caused by work on the system that is not described in this manual.

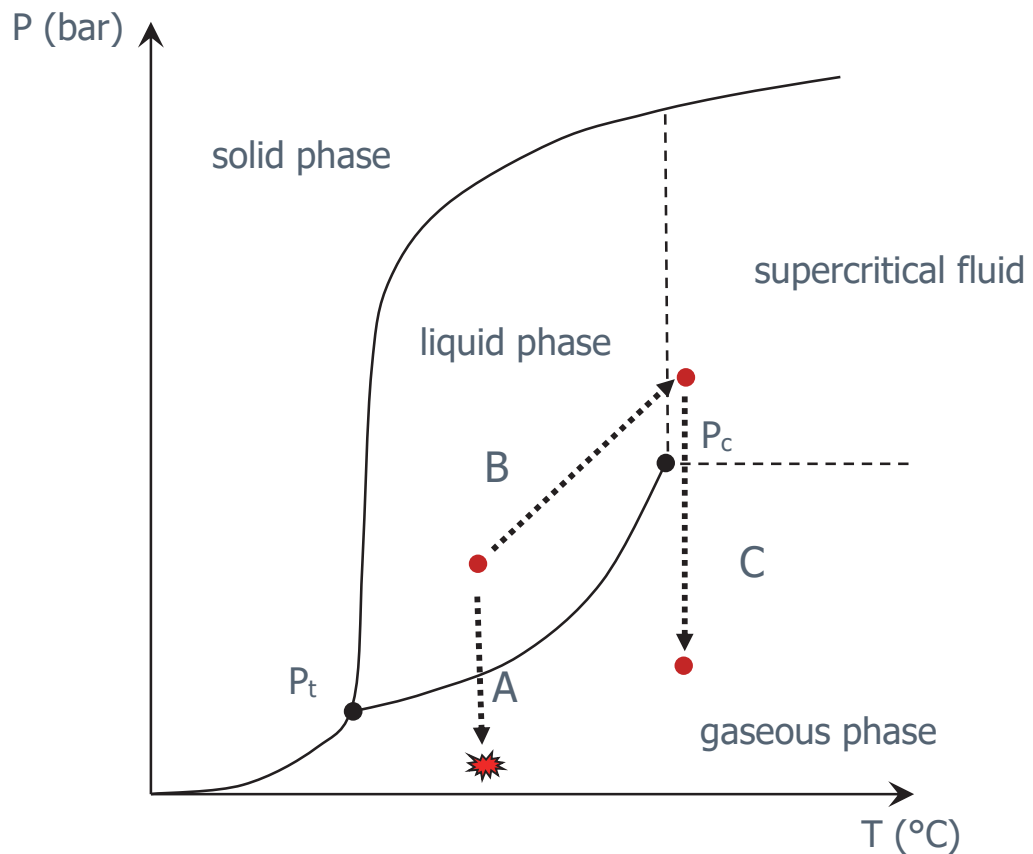
4. Method

4.1 Critical Point Drying Method

Drying water-containing biological and material specimens in air or under vacuum can drastically alter their structures or even destroy them completely by tangential forces, caused by surface tension of the water (A).

The surface tension is a quality of surfaces between a liquid and a gas and the tangential forces appears by boundary crossing from liquid to gaseous phase.

Phase diagram



P_c = Critical Point

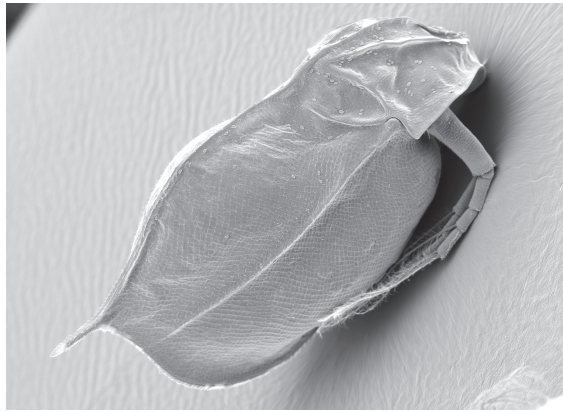
P_t = Triple Point

A = Air drying (phase boundary crossing)

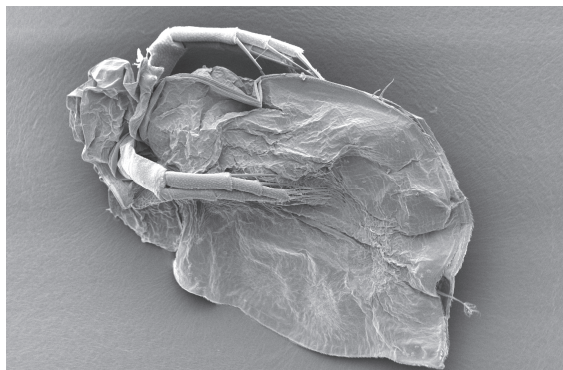
B and C = Critical point drying (no phase boundary crossing)

For biological and most micro-electro-mechanical (MEM) samples, critical point drying cannot be performed with water. Water's critical point is at 374°C and 228.5bar. Every biological and MEM sample would be destroyed at this temperature and pressure. Therefore, the use of CO₂ as transitional fluid is the first choice for critical point drying. It is liquid at room temperature at a comparative low pressure of 60bar. Its critical point is at 31°C and 73.8bar. Technically these temperature and pressure requirements of the CO₂ can be implemented relatively easily. Hence, the water in the cell is replaced by an exchange medium like acetone or ethanol, which unlike water, are very soluble with liquid CO₂. After the replacement of the water by an exchange fluid, this exchange medium is substituted with liquid CO₂ through serial dilution steps. Thereafter by increasing the temperature, the pressure is increased proportionally and will transfer the CO₂ through its critical point into a supercritical state (B). At constant temperature the controlled and careful depressurization converts the supercritical CO₂ into its gaseous phase without crossing the phase boundary between liquid and gas (C). After coating, the dried sample can be analyzed in a scanning electron microscope (SEM).

Comparison between air and critical point drying:



Critical point dried sample (Water flea)



Air dried sample (Water flea)

5. Description and Specification

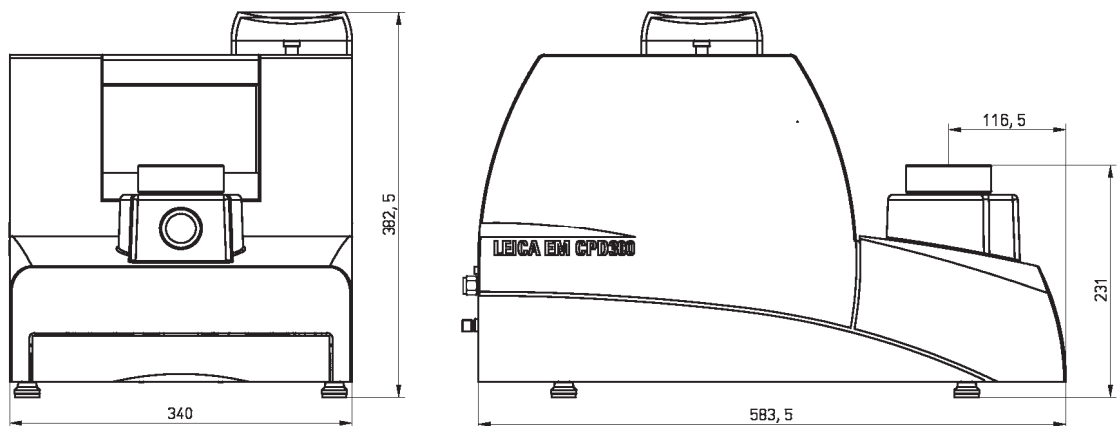
Introduction

Biological specimens intended for investigation in a scanning electron microscope must be dried before being introduced into the microscope to allow the specimen to be imaged. If specimens with water content are dried, tangential forces, caused by surface tension of the water, destroys the structures of the specimen as mentioned in the previous chapter.

Critical point drying with the EM CPD300 is the optimal method to prevent the damaging effects of the above-mentioned forces by bringing the liquid to the gas phase without boundary crossing.

5.1 Technical specifications

Dimensions:



Weight:

Empty weight 31 kg

Sample chamber:

Chamber dimension Ø 60 mm x 62 mm
Volume 185 ml in empty chamber

Pressure:

Safety bursting membrane 105 bar at 20 °C (+/-10%)
Software controlled cut off 80 bar
Max. operating pressure 79 bar
(Software controlled)

Temperature:

Heating range adjustable 33 °C to 43 °C
Software controlled cut off >45 °C
Heating time Slow 1 °C/min.
Heating time Medium 2 °C/min.
Heating time Fast 3 °C/min.

Cooling

Cooling capacity	90 W at -5°C (2500 1/min)
Refrigerant	R134a
Quantity	120 g
Cooling range	adjustable 5 °C to 25 °C
Cool down time	2.0 °C/min.

Electrical Connection:

Voltage	100 V -230 V
Frequency	50 / 60 Hz
Power consumption	170 VA

Gas Connection:

Gas inlet	M 12 x 1.5
Gas outlet	Ø 8 mm (inner diameter)

Requirements

Transitional fluid: Carbon Dioxide (chemical formula CO₂)
CO₂ pressure bottle with feed pipe
Exchange fluid: Acetone, Ethanol

6. Preparation of Specimens

Wash in a physiological salt solution.

Fix chemically with a suitable fixing agent.

Wash out fixing agent with a suitable buffer solution.

Dehydrate the chemically fixed specimen with acetone or ethanol.

(Protocol examples are shown in the EM CPD300 Application booklet).

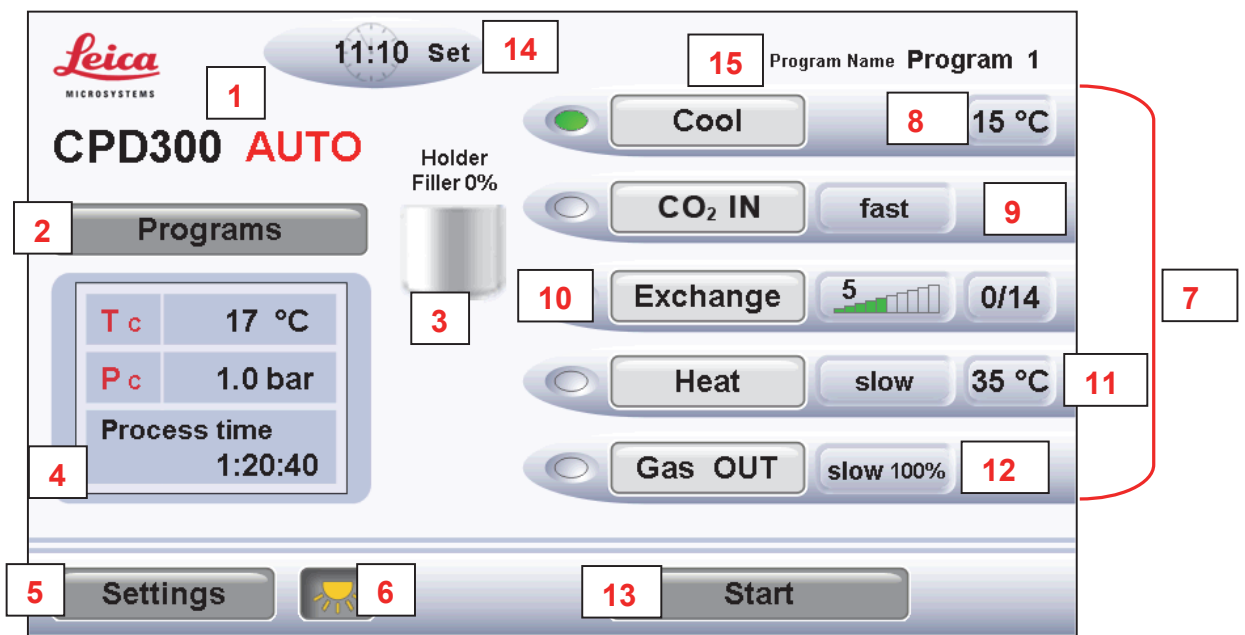
7. Automatic Operation of EM CPD300

7.1 Screen descriptions

Initialization screen: Touch screen after initialization.



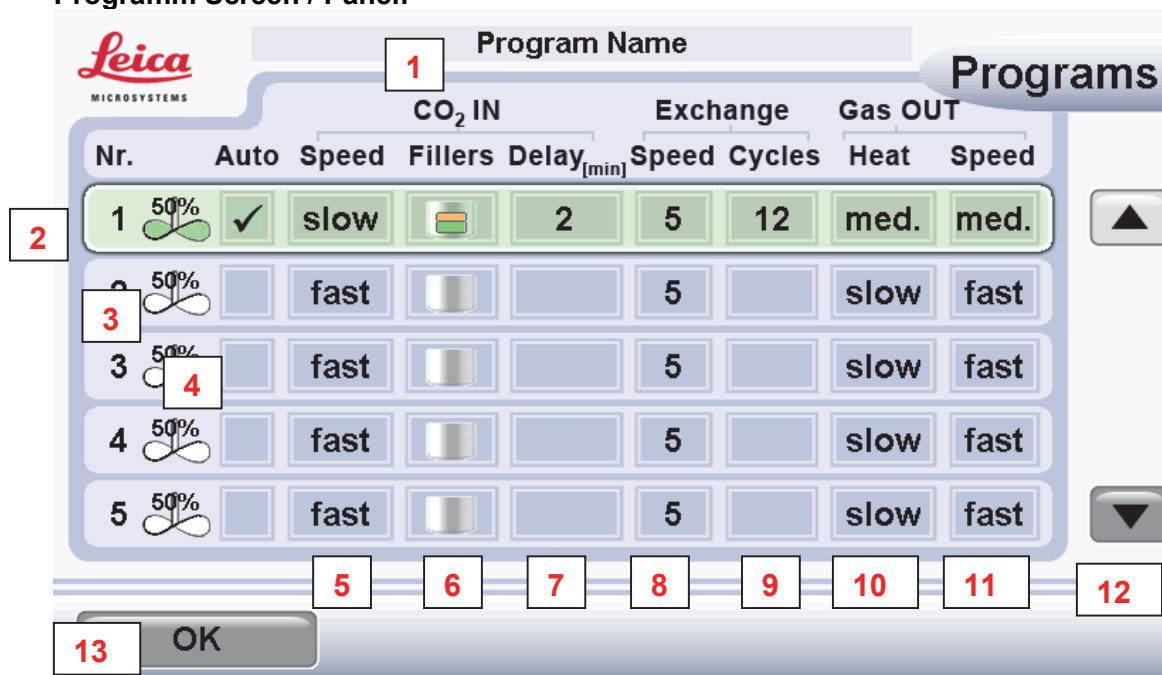
Main Screen:



Dark gray buttons can be activated, light gray buttons are inactive!

- 1 Version of the EM CPD300.
- 2 Switch to program panel (see page 15).
- 3 Status display of fillers and holder in the sample chamber.
Programmable under programs.
- 4 Status display temperature, pressure and time to finish the process
(last point only with auto function).
- 5 Switch to settings (see page 37).
- 6 Light on/off
- 7 Status display of programmed process.
In auto version buttons have no function.
- 8 Cooling temperature to keep CO₂ fluid (can be changed under settings, page 37).
- 9 CO₂ influx speed in pressure chamber.
Programmable under programs.
- 10 Exchange speed (1-10) and status of finished exchange cycles.
Programmable under programs.
- 11 Heating speed and heating temperature for critical point.
Programmable under programs.
- 12 Status display gas out speed.
Programmable under programs.
- 13 Process start. (after defining program).
- 14 Timer function (see page 27).
- 15 Program name of activated program (see page 15).

Programm Screen / Panel:



- 1 Activates key pad to enter program name.
- 2 Activated program is marked green.
- 3 Stirrer on / off with speed control.
- 4 Activation of auto version. If not highlighted manual version is active. Only selectable in automated version.
- 5 Sets speed of CO₂ influx in pressure chamber. Three possibilities: slow, medium, fast.
- 6 Switch to filler and holder panel. Display of filler and holder status.
- 7 Sets delay time after influx of CO₂ and before starting exchange process.
- 8 Sets exchange speed from 1-10.
The number relates to the setting of the needle valve during exchange. 1 means a small opening and staying open for longer (slow flowing speed), 10 means a big opening for a short amount of time (fast flowing speed). The parameters where set in a way that after a minimum of 12 exchange cycles only liquid CO₂ is left in the chamber.
- 9 Sets exchange cycles. 12 cycles means one chamber volume is completely exchanged. Minimum is 12 cycles.
- 10 Sets heating speed for critical point. Three possibilities: slow, medium, fast.
- 11 Sets gas out speed. Possibilities: slow, medium, fast.

Slow speed can be decreased up to 20% of its normal speed.

12 Scrolls programs from 1-10.

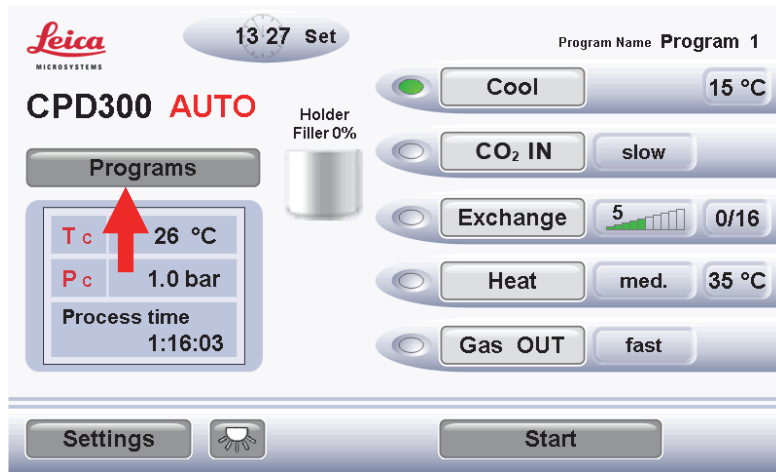
13 Confirms activated program. Switch to main screen.

Filler/Holder Panel:

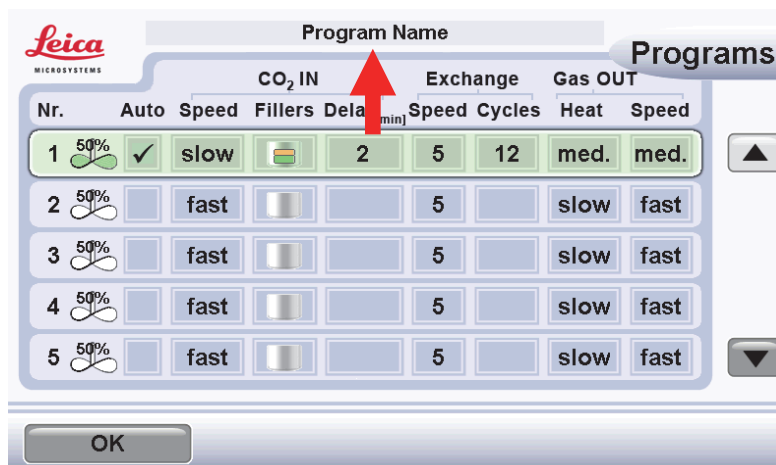


- 1 Filler and holder panel.
- 2 Status display of fillers and holders.
- 3 Sets specific holder and fillers. Combination of holders and fillers depends on their volumes.
- 4 Confirms filler and holder setting.

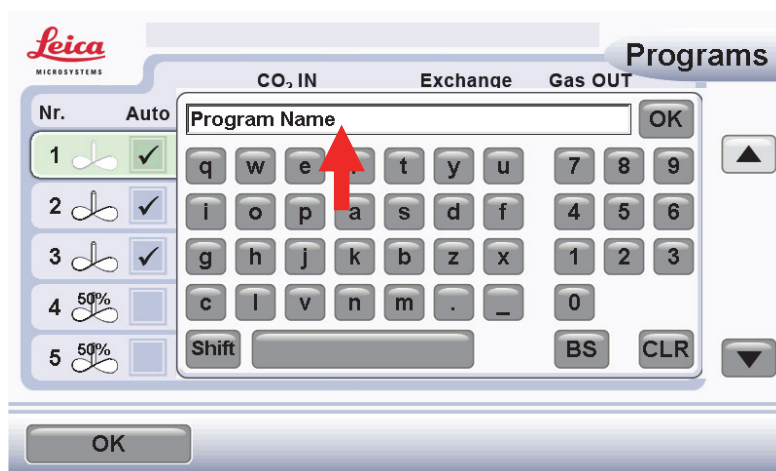
7.2 Programming by example



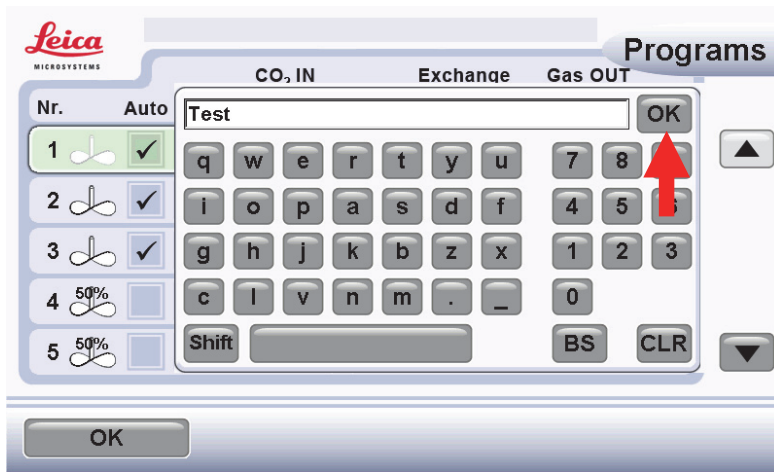
Press Programs



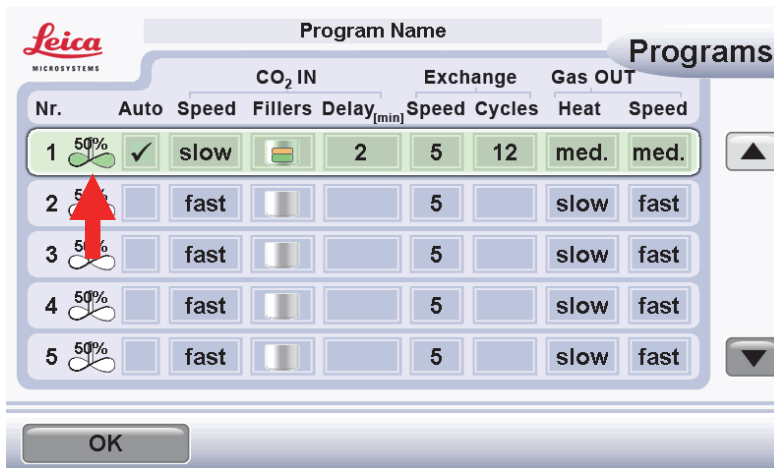
Press Program Name



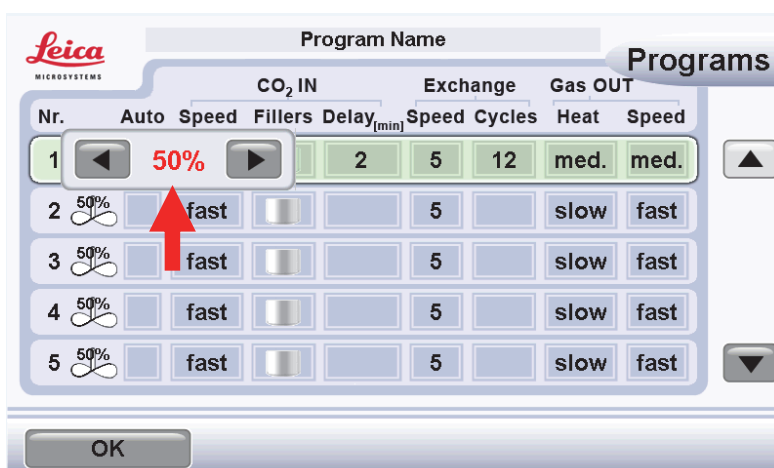
Type Program name



Press OK to confirm program name

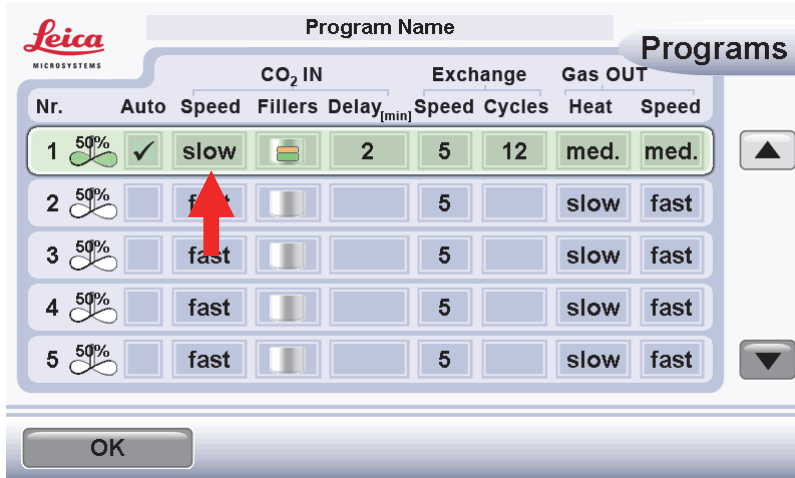


Activate stirrer

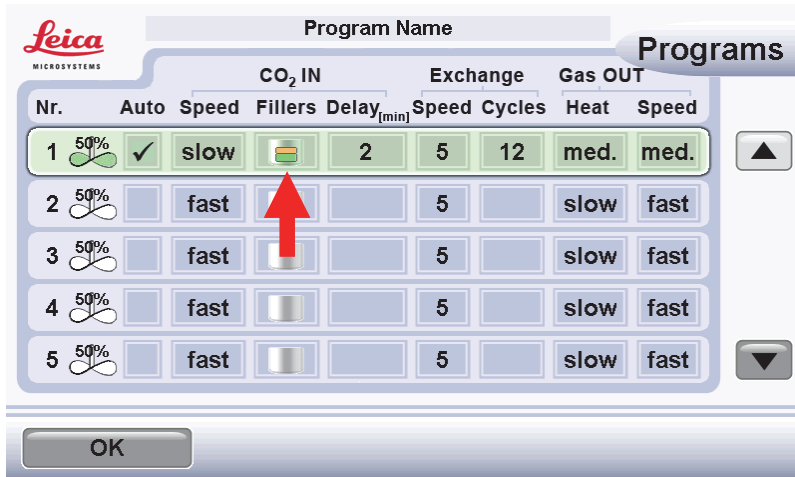


Select stirrer speed

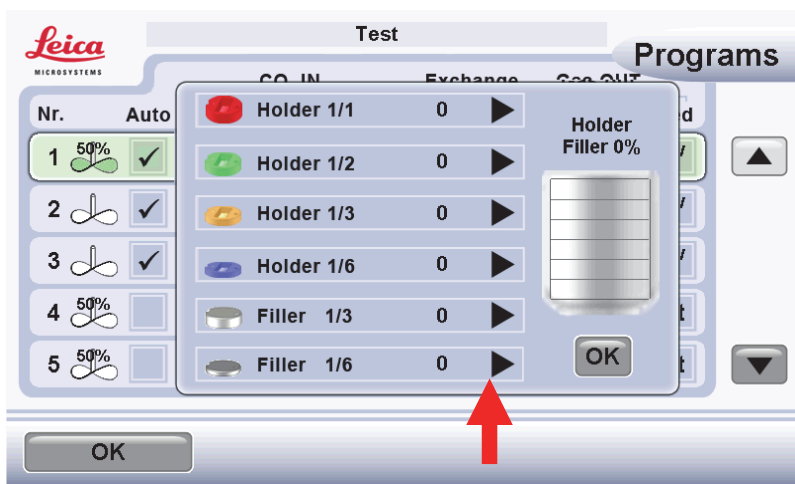
Speed display will disappear after 4 sec.



Activated stirrer symbol becomes green
Set CO₂-IN speed

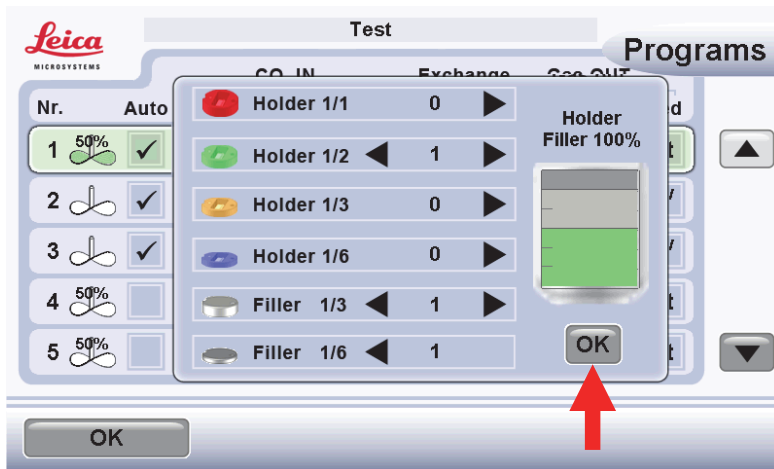


Press Fillers

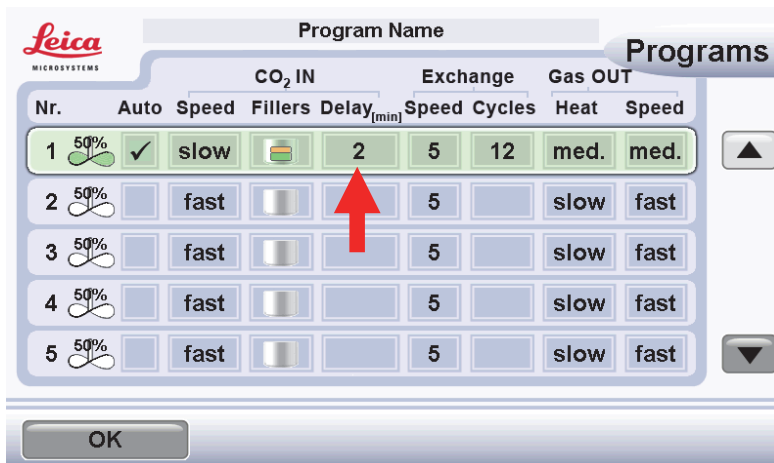


Program Filler/Holder combination

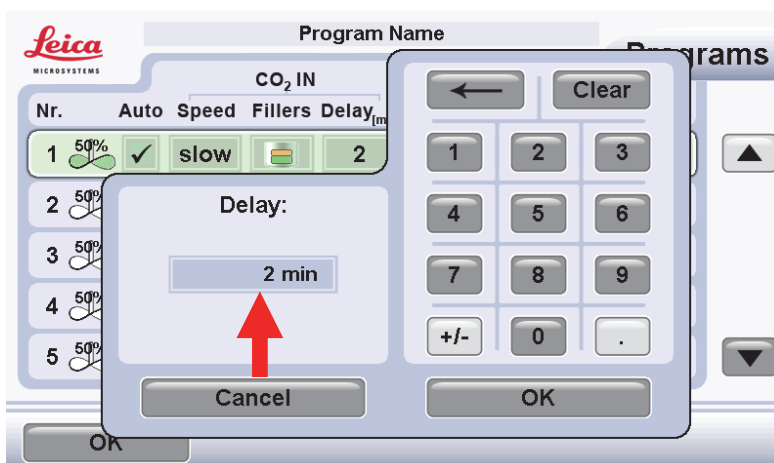
Example:
1/2 Holder with 1/6 and 1/3 Filler



Press OK to confirm Filler/Holder settings

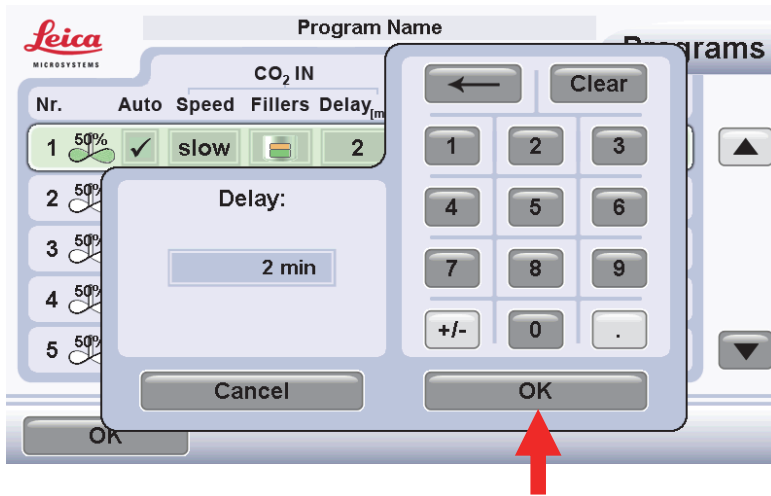


Press Delay

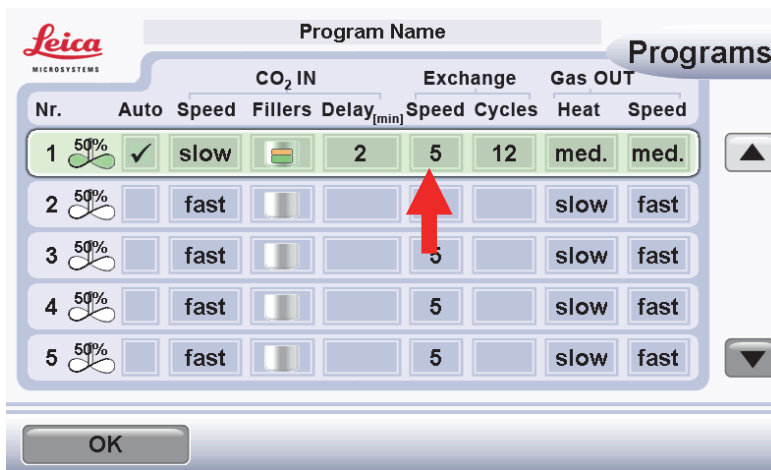


Set Delay time

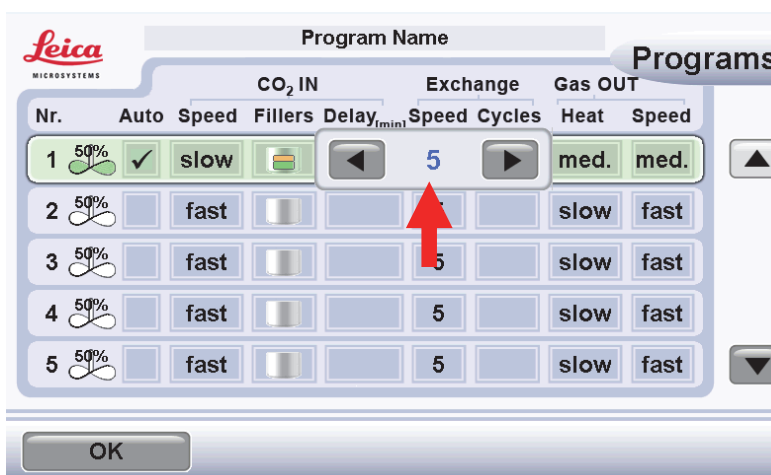
Example:
Set to 2min



Press OK to confirm



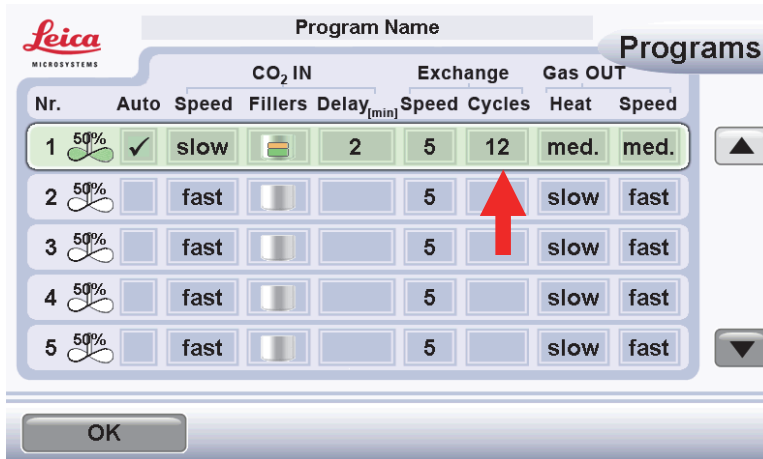
Press Exchange Speed



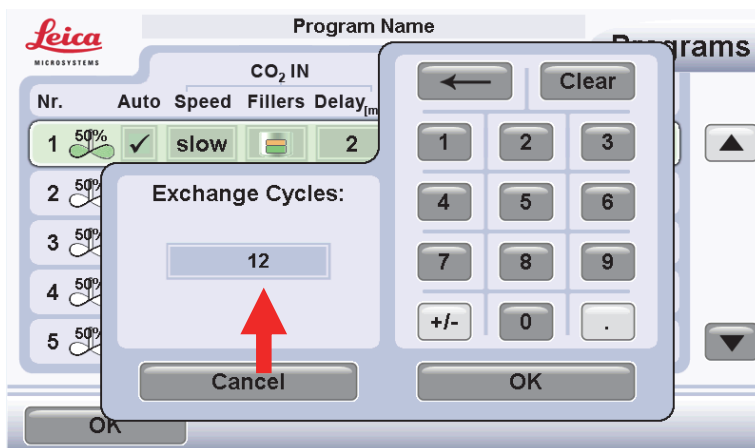
Set Exchange Speed

Example:
set to 5

Exchange Speed display will disappear after 4 sec.

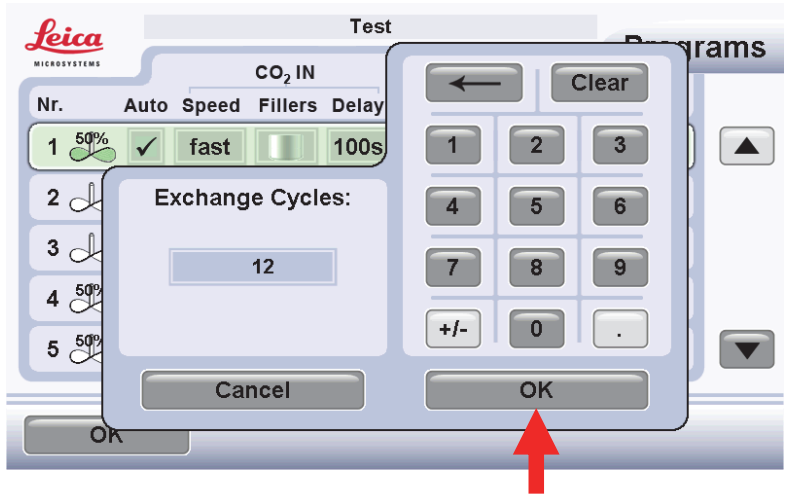


Press Exchange Cycles

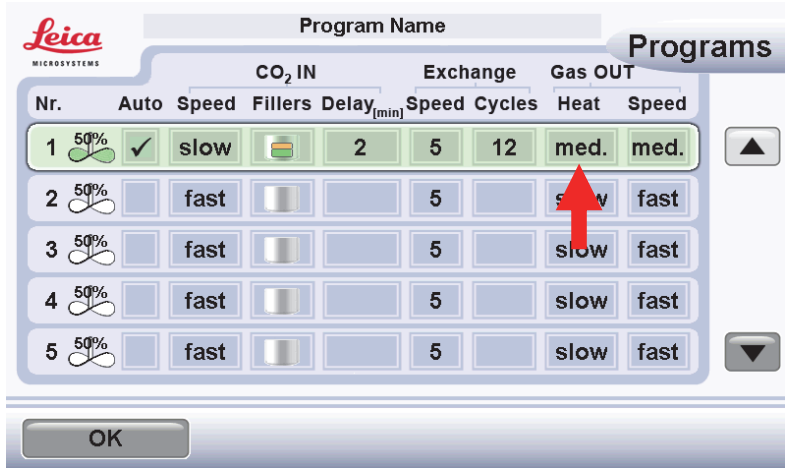


Set Exchange Cycles

Example:
set to 12

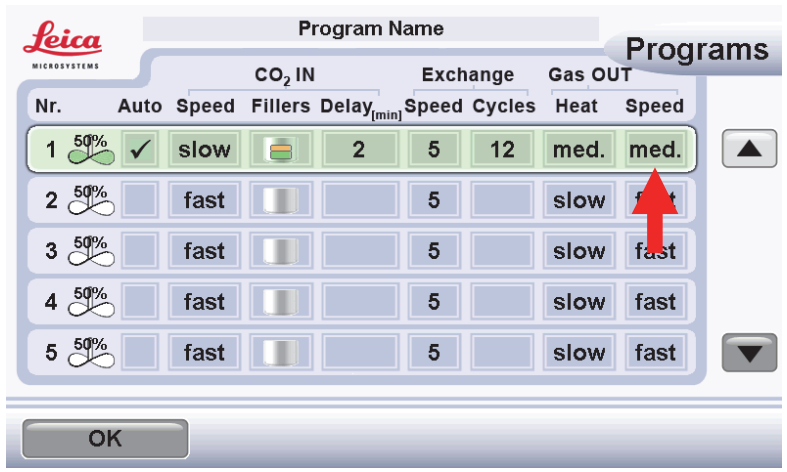


Press OK to confirm



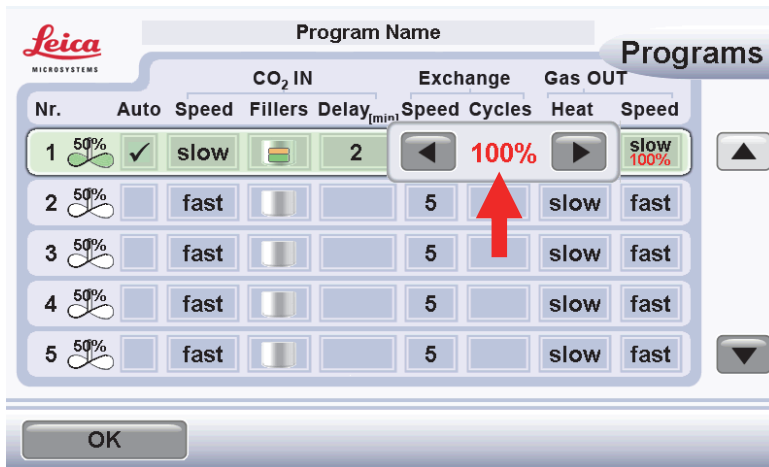
Set heating speed

Example:
from medium to fast



Set Gas out speed

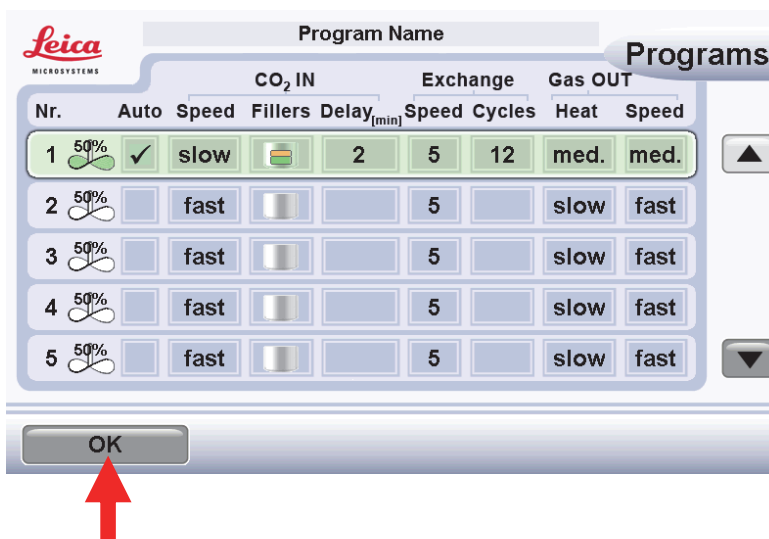
Example:
from fast to slow



Select slow speed setting

Example:
Set to 100% slow

Slow Gas out Speed display will disappear after 4 sec.



Press OK to confirm



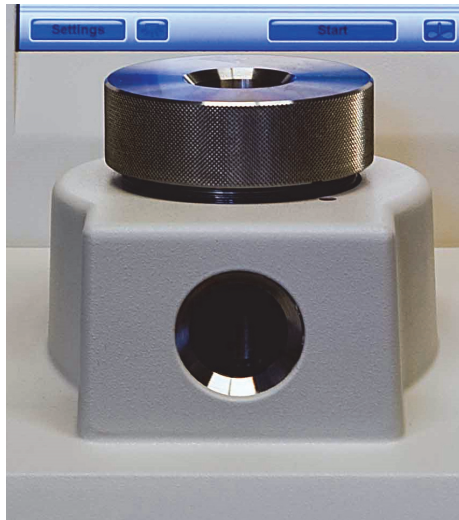
Press Start to activate program Test

7.3 Programming the EM CPD300 procedure

Before placing the sample into the pressure chamber, program the EM CPD300 protocol. Switch to the program screen panel and follow the instructions under Program Panel (see page 17-25). Do not forget adjustment of the pressure threshold for the bottle empty function (see page 38-39).

7.4 Filling the Sample Chamber with exchange fluid

Open the screw-on cover of the sample chamber by turning cover counter clockwise.



Sample Container with quick release pin

Before filling the sample chamber with exchange fluid, place the magnetic stirrer rod in bottom of the chamber.

Fill exchange fluid into the sample chamber until the stirrer is covered.

Then fill the sample container with exchange fluid to such a level that the sample holder and specimen will be completely submerged when inserted.

7.5 Inserting Specimen into Sample Container

Open lid and transfer prepared specimen into a suitable sample holder. The sample holder must be filled with exchange fluid prior to transfer.

Insert the sample holder into the sample container and fill sample container with exchange fluid until the sample holder is covered. Transfer the sample container into the sample chamber and check if sample holder is still covered with exchange fluid.

Adjust container volume if required with fillers or additional sample holders.

If sample holders with the volume of 33 – 50% are used, more sample holders or fillers can be added to the pressure chamber (up to 100%).

Fully open the shut-off valve of the CO₂ bottle.

7.6 Preset of Cooling Temperature of the Sample Chamber

The pressure chamber cools up to the preset cooling temperature (15°C factory setting). The cooling temperature depends on the bottle temperature. Please see chapter “7.6.1. CO₂-Bottle Temperature / Pressure Function” to select the appropriate cooling temperature. The cooling unit automatically keeps the temperature at the preset cooling temperature or below (see subchapter 7.6.1 below).

7.6.1 CO₂-Bottle Temperature / Pressure Function

For correct filling of the pressure chamber with CO₂ a temperature difference of 4 °C minimum and a pressure difference of 5 bar is essential. Therefore, the pressure chamber has always to be minimum 4 °C cooler than the CO₂-Bottle (see list below). You can find the adjustment of pressure chamber temperature under “settings” (see operating manual).

The factory preset cooling temperature of the pressure chamber is 15 °C. If the CO₂ does not fill the chamber within a certain time, “Timeout CO₂-IN” shows in the yellow box. If the poral filter is clean and the bottle is not empty the reason for the warning is the CO₂ temperature bottle which is cooler than the chamber temperature. This means, due to the low temperature difference, the pressure of the CO₂ in the bottle is not sufficient to fill-up the chamber.

The temperature of the bottle can be estimated by measuring the bottle surface with a thermometer. The CO₂ temperature is then about 2 °C cooler than the bottle surface. Decrease the chamber temperature according to the list below and fill again. The green marked values indicate the optimal working temperature and pressure range.

Example: If the bottle surface temperature is 22 °C the estimated CO₂ temperature is 20 °C, the cooling temperature of the chamber should be set to 15 °C.

CO ₂ -Temperature (°C)	Recommended pressure chamber cooling temperature (°C)
14	9
15	10
16	11
18	13
20	15
22	17
24	19
25	20
26	21
28	23

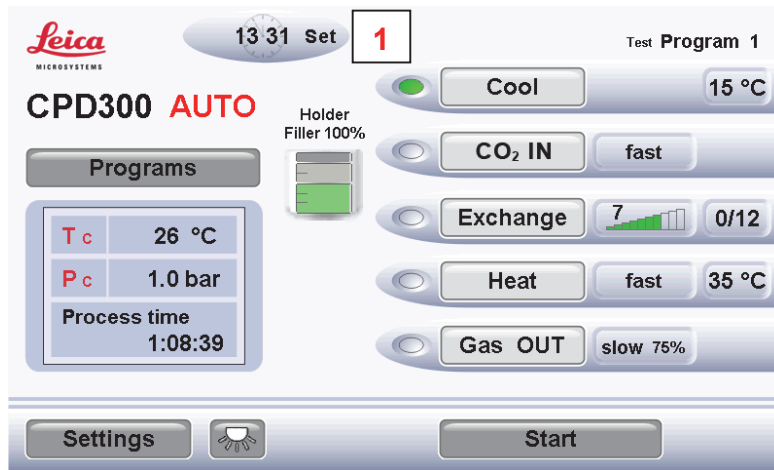
Fully open the shut-off valve of the CO₂ bottle.

7.7 Starting the EM CPD300 auto procedure

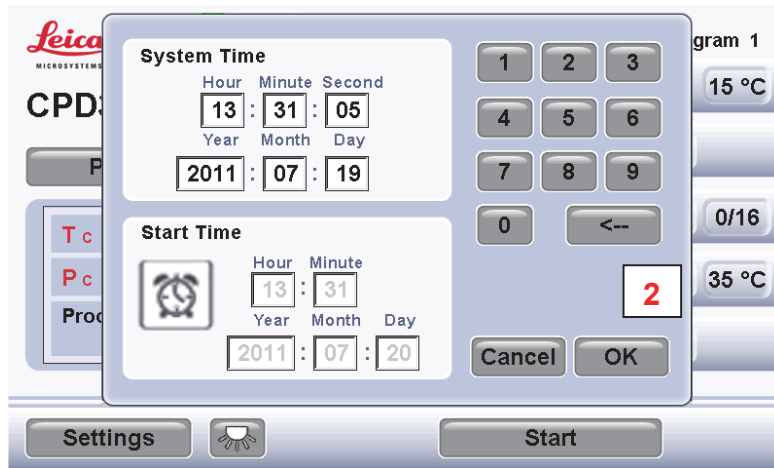
After programming go back to the main menu and press start.

7.8 Time and timer function

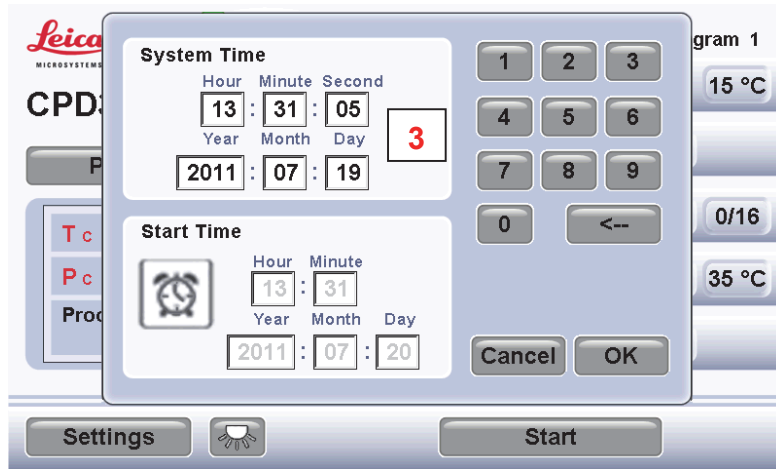
Press timer function (1).



The timer display pops up (2).



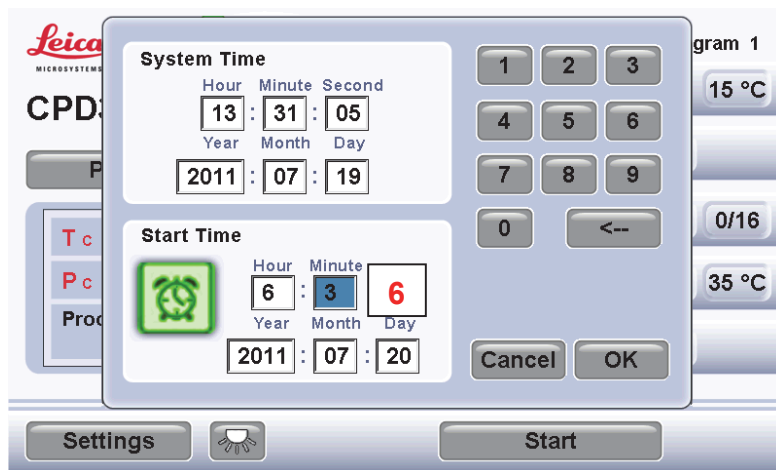
Now you can change the local time (3)...



...or set the timer with the keypad (4). The timer function is activated if watch symbol is green (5).



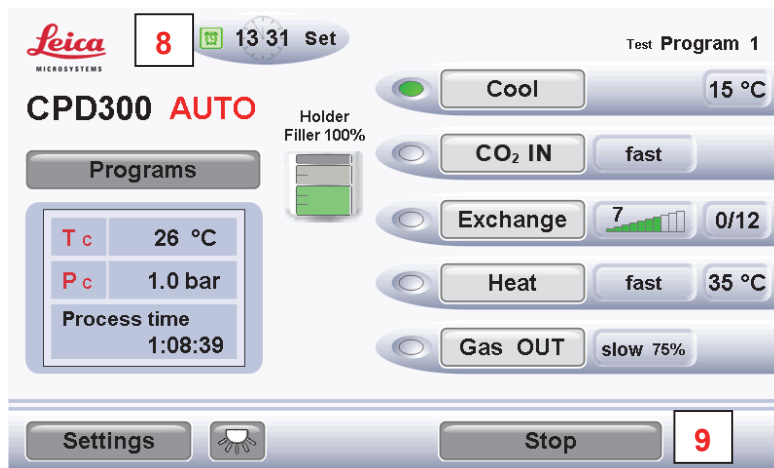
Activated time fields become blue (6).



Press OK and settings will be saved (7).

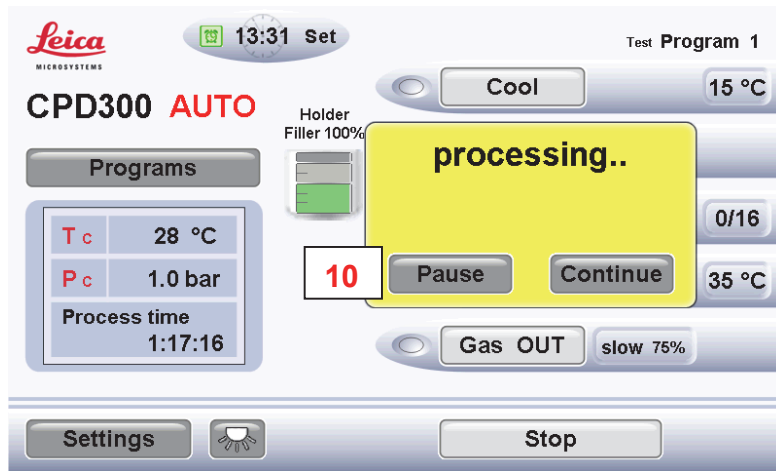


In the main screen the green watch symbol beside the local time indication shows that time is activated (8). By activating the timer no programming or other changes are possible.

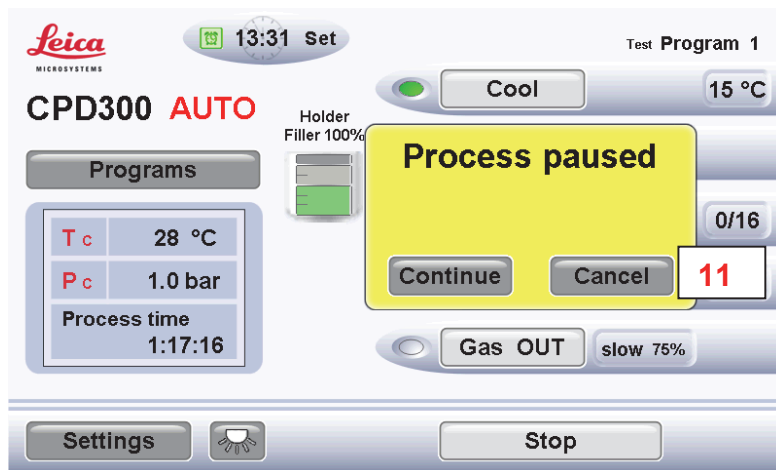


To cancel timer settings, press stop (9) ...

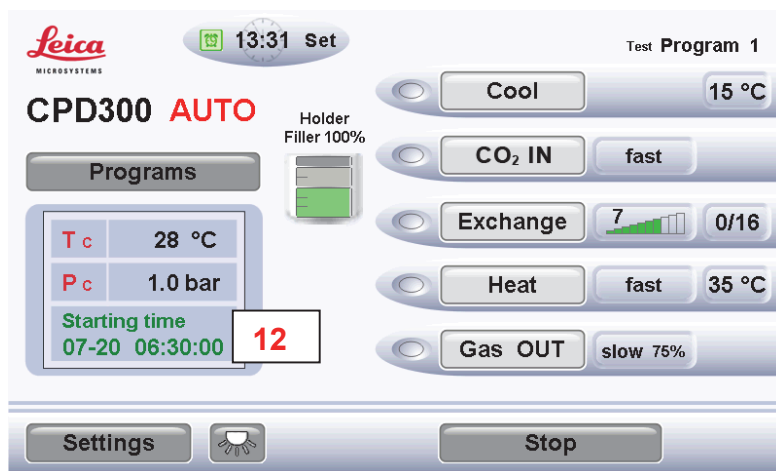
...then pause (10)...



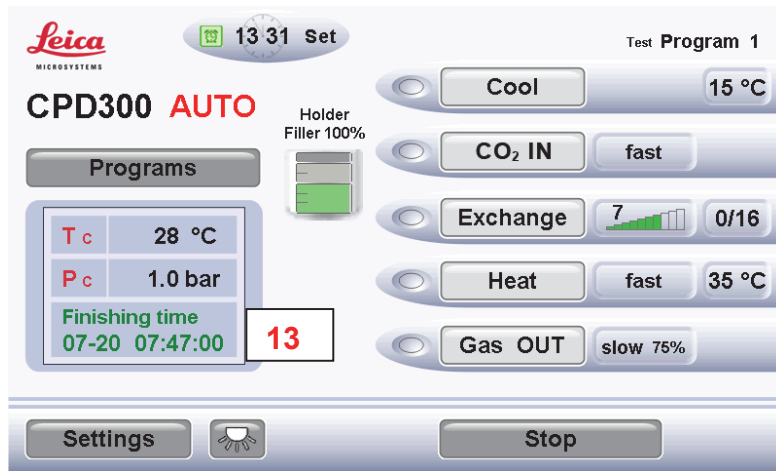
...and cancel (11).



Touch Process time field to check (in the main screen) the timer starting time (12)...



...and touch starting time to check finishing time (13).



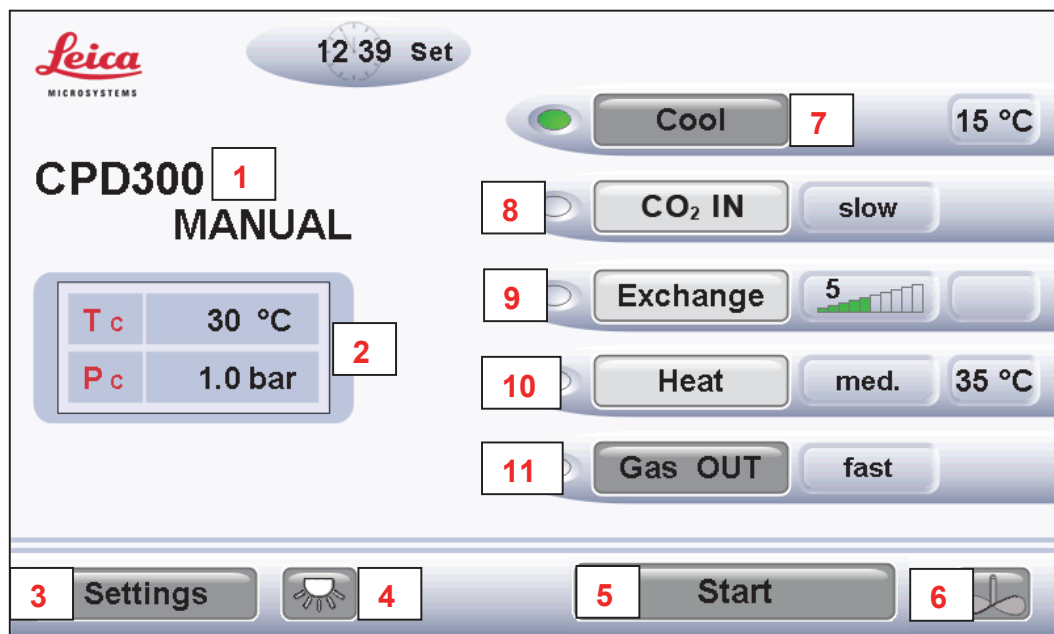
8. Manual Operation of EM CPD300

8.1 Screen descriptions

Initialization screen: Touch screen after initialization

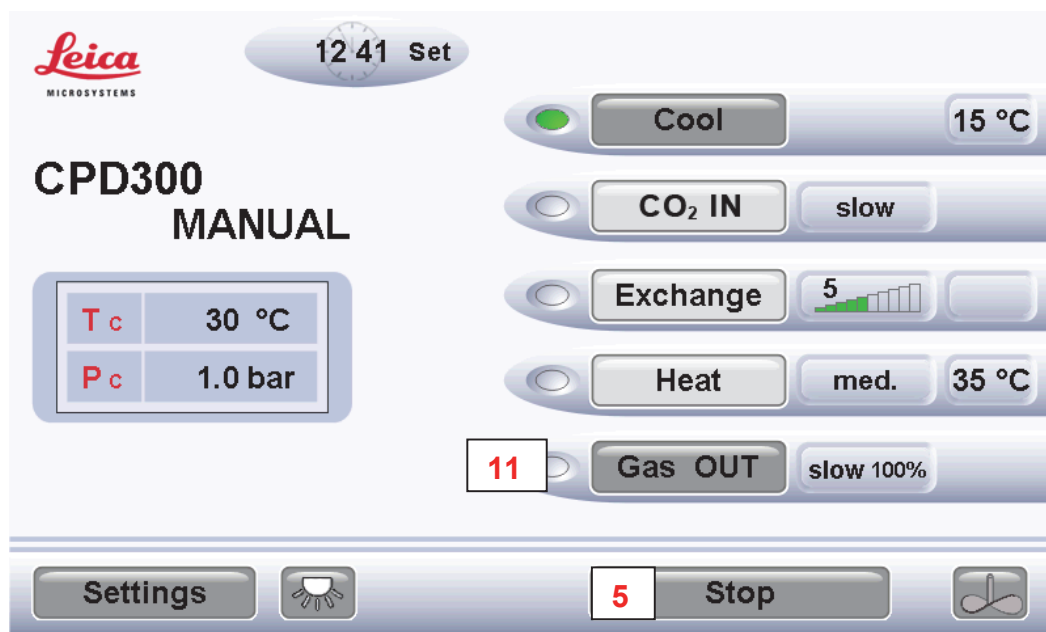


Main Screen before start:



Dark gray buttons can be activated, light gray buttons are inactive!

Main Screen after start:

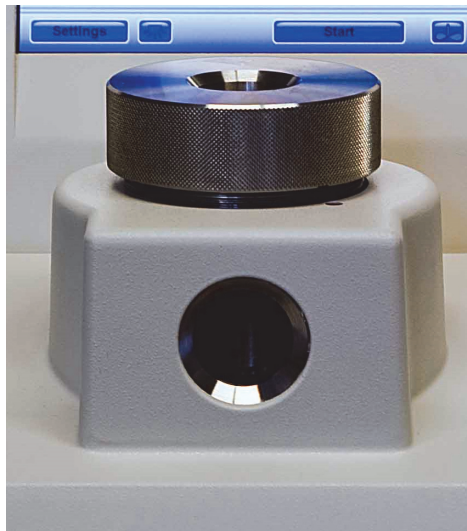


- 1 Version of the EM CPD300.
- 2 Status display temperature, pressure.
- 3 Switch to Settings (see Page 37).
- 4 Light on/off.
- 5 Process Start / Stop.
- 6 Stirrer on / off. Is controlled in setting panel (see Page 37).
- 7 Cooling temperature to keep CO₂ fluid.
- 8 Sets speed of CO₂ influx in pressure chamber.
Three possibilities: slow, medium, fast. To inactivate press again.
- 9 Sets exchange speed. Speed from 1-10. Shows opening status of the needle valve. To inactivate press again.
- 10 Sets heating speed for critical point. Three possibilities: slow, medium, fast.
To inactivate press again.
- 11 Sets Gas out speed. Possibilities: slow, medium, fast. Slow speed can be decreased up to 20% of its normal speed. Adjustment under Settings / advanced slow Gas Out (see page 37).
- 12 Sets Time (see page 27).

8.2 Filling the chamber with exchange fluid

Do not forget adjustment of the pressure threshold for the bottle empty function (See page 38-39)!

Open the screw-on cover of the sample chamber by turning counter clockwise.



Spacer with magnetic Stirrer

Before filling the sample chamber with exchange fluid, place the magnetic stirrer rod together with the spacer into bottom of the sample chamber.

Fill exchange fluid into the sample chamber until the stirrer and spacer is covered.

Fill the sample chamber with exchange fluid to such a level that the specimen holder with specimen will be completely submerged when inserted.

8.3 Inserting of Specimen into Sample Chamber

Open lid and transfer prepared specimen into a suitable sample holder.

Quickly insert the sample holder with the specimen into the sample chamber filled with exchange fluid. Check if sample holder is still covered with exchange fluid.

8.4 Cooling of the Sample Chamber

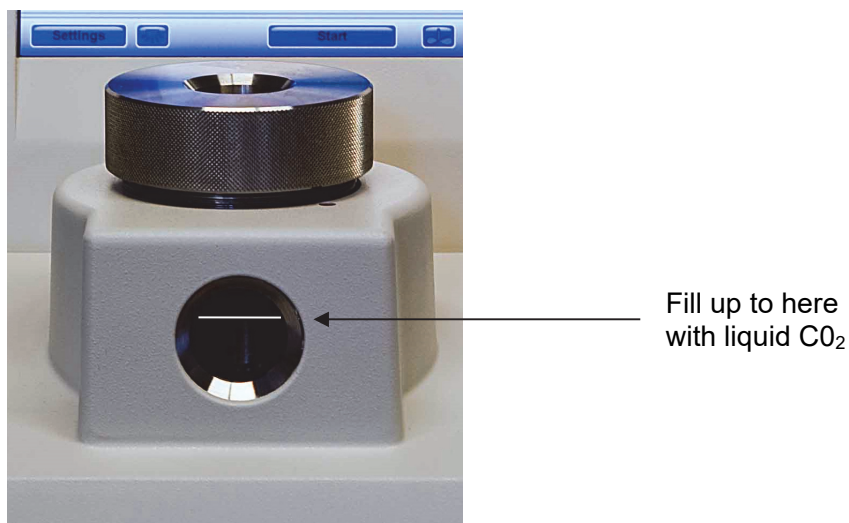
After tightly closing the screw-on cover of the sample pressure chamber, press Start. Press the cooling button and wait until pressure chamber cools up to the preset cooling temperature (15°C factory setting). The cooling temperature depends on the bottle temperature. Please see sub chapter "7.6.1 CO₂-Bottle Temperature / Pressure Function" to select the appropriate cooling temperature. The cooling unit automatically keeps the temperature at the preset cooling temperature or below.

8.5 Filling the CO₂ into Sample Chamber

Wait until the sample chamber temperature has reached 15°C. Once this temperature is reached, completely open the shut-off valve of the CO₂ bottle. Press CO₂ in with the selected speed (slow, medium, or fast).

The pressure display shows the pressure in the sample chamber (identical to the pressure in the CO₂ bottle).

Fill the sample chamber up to the upper edge of the front viewing glass with liquid CO₂.



Press the CO₂-In button again. CO₂ goes off. The CO₂ level in the sample chamber may rise slightly.

8.6 Mixing the two liquid media

Press the STIRRER key. The magnetic stirrer is switched on. The magnetic stirrer should not be used if the specimens are very delicate.

8.7 Exchange media mixture from Sample Chamber

Press the EXCHANGE button. Drain the media mixture from the sample chamber until the specimen is barely covered by the liquid.

Press the EXCHANGE button again to close.

This procedure of filling, mixing, and draining needs to be repeated several times (minimum 12 times) before heating to remove all the exchange fluid from the specimens. Multiple and, or larger specimens require more changes.

8.8 Heating the Sample Chamber

Press the HEAT button and wait until sample chamber is heated up to 35 °C. The cooling unit automatically keeps the temperature of the pressure chamber at 35 °C. With the rising temperature the pressure in the sample chamber also increases as shown on the display.

At the time the critical temperature and the critical pressure (31 °C and 73,8 bar for CO₂) have been passed, the liquid CO₂ is transformed into the supercritical status. The system will stop at 35 °C and 79 bar to be safe on the supercritical state. The change of the physical state can be observed through the front viewing glass.

8.9 Releasing gaseous CO₂ from Sample Chamber

After reaching the supercritical status, choose the GAS-OUT speed (slow, medium or fast) and press the GAS-OUT button. The temperature will be constant at 35 °C only when the valve is opening to release the CO₂, which will change the physical status from supercritical to gaseous due to the pressure decrease under constant temperature.

9. Removing Dried Specimen



Close the shut-off valve of the CO₂ bottle.

Open the screw-on cover of specimen sample chamber. Remove specimen holder with dried specimen from sample chamber for further processing.

Since the dried specimen is highly hygroscopic, it has to be coated as quickly as possible with a thin metal- or carbon film to protect it from atmospheric humidity.

If this is not possible, it is recommended to keep the specimen in a desiccator until it is processed further.

10. Switching off the Unit

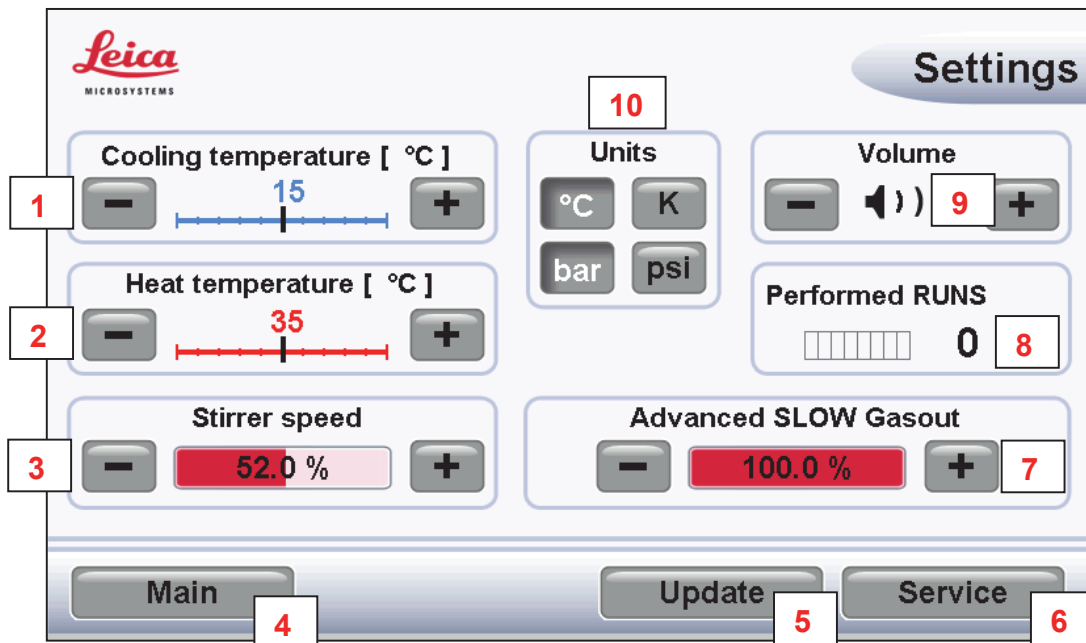


Leave the sample chamber open. Leave the CO₂ bottle closed.

Switch off the main switch located on the unit.

11. Setting Panel

Dark gray buttons can be activated, light gray buttons are inactive!



- 1 Sets cooling temperature to keep CO₂ fluid. (Not recommended to change).
- 2 Sets heating temperature to reach the critical point. (Not recommended to change).
- 3 Sets stirrer speed, (recommended adjusting to sample sensitivity).
- 4 Press Main to switch to main screen.
- 5 Press update for software updates (USB).
- 6 Service Panel just for Service technician (see Service Manual).
- 7 Adjustment of Gas-out speed at slow. Function under settings only in the manual version accessible. In the auto version the advanced slow Gas-out speed adjustment is accessible under program display (see page 15).
- 8 System counts every complete run for maintenance advices (see page 40).
- 9 Volume change.
- 10 Physical unit change.

12. CO₂-Bottle Temperature / Pressure Function

For correct filling of the pressure chamber with CO₂ a temperature difference of 4 °C minimum and a pressure difference of 5 bar is essential. Therefore, the pressure chamber has always to be minimum 4 °C cooler than the CO₂-Bottle (see list below). You can find the adjustment of pressure chamber temperature under “settings” (see operating manual).

The factory preset cooling temperature of the pressure chamber is 15 °C. If the CO₂ does not fill the chamber within a certain time, “Timeout CO₂-IN” shows in the yellow box. If the poral filter is clean and the bottle is not empty the reason for the warning is the CO₂ temperature bottle which is cooler than the chamber temperature. This means, due to the low temperature difference, the pressure of the CO₂ in the bottle is not sufficient to fill-up the chamber.

The temperature of the bottle can be estimated by measuring the bottle surface with a thermometer. The CO₂ temperature is then about 2 °C cooler than the bottle surface. Decrease the chamber temperature according to the list below and fill again. The green marked values indicate the optimal working temperature and pressure range.

Example: If the bottle surface temperature is 22 °C the estimated CO₂ temperature is 20 °C, the cooling temperature of the chamber should be set to 15 °C.

CO ₂ -Temperature (°C)	Recommended pressure chamber cooling temperature (°C)
14	9
15	10
16	11
18	13
20	15
22	17
24	19
25	20
26	21
28	23

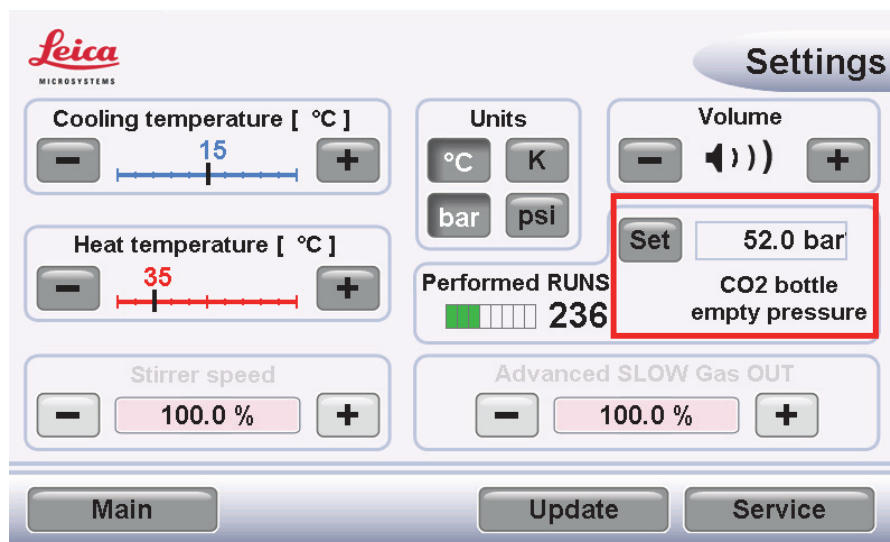
13. Adjustments of Pressure Threshold for Bottle Empty Function

The bottle empty function was developed to protect the samples if the CO₂ bottle becomes empty during a run. When the warning occurs, all valves will be closed so that the pressure chamber is sealed, and the empty bottle can be exchanged with reduced possibility of sample damage. The threshold for this function has to be adapted to the CO₂ temperature. See list below. Green marked values indicate optimal working temperature and pressure range.

CO ₂ -Temperature (°C)	Recommended threshold for pressure (bar)	Pressure of full CO ₂ -Bottle (bar)
14	47	50
15	48	51
16	49	52
18	52	55
20	54	57
22	57	60
24	60	63
25	61	64
26	63	66
28	66	69

Adjustments of Pressure Threshold:

Go to settings and set the desired empty pressure threshold (see table above) under the “CO₂ bottle empty pressure” area.

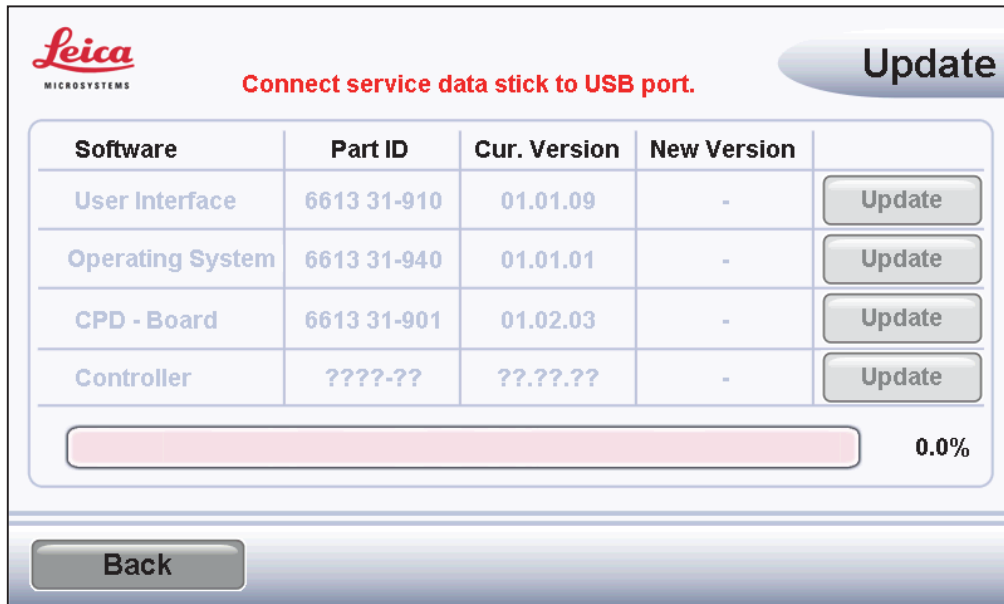


Change CO₂ bottle empty pressure threshold value according to the list on page 45. The CO₂ temperature can be estimated by measuring bottle surface with thermometer. CO₂ temperature is then about 1-2 °C cooler than the bottle surface.

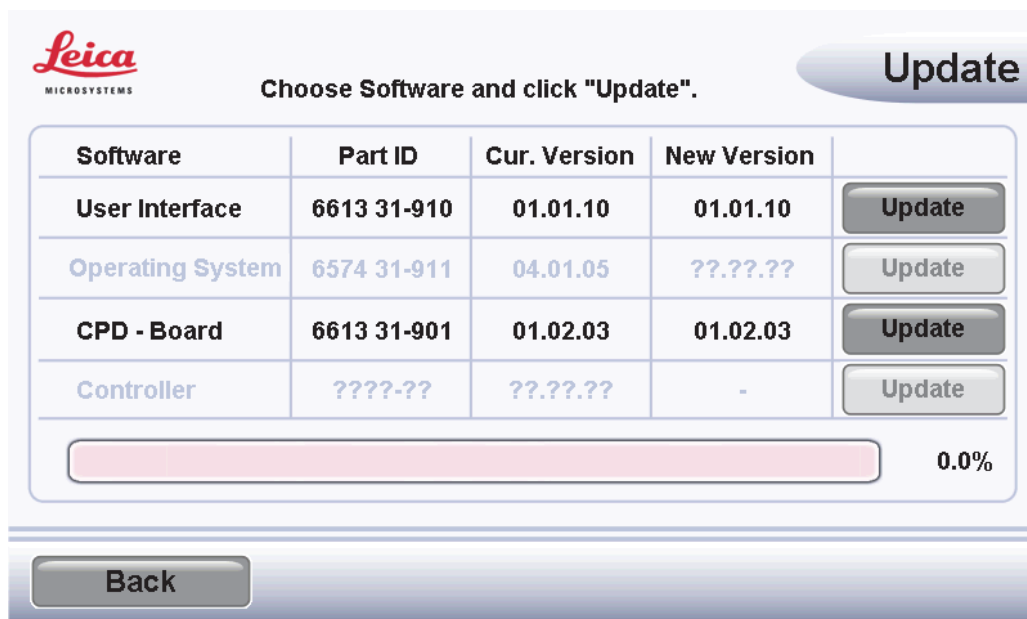
Press „ Update “ to confirm.

14. Software Update

Insert USB stick with EM CPD300 software.



Software modules will be automatically recognized by the system. Choose the module you want to update.



After software update, press back.

Leica
MICROSYSTEMS

Updating Chamber Controller...

Update

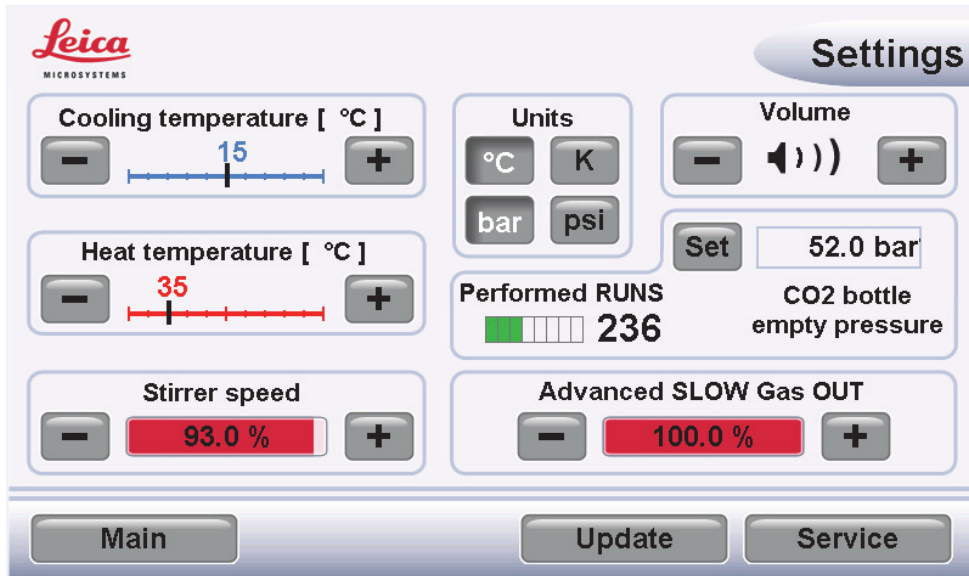
Software	Part ID	Cur. Version	New Version	
User Interface	6613 31-910	01.01.09	01.01.09	Update
Operating System	6613 31-940	01.01.01	???.??.??	Update
➔ CPD - Board	6613 31-901	01.02.03	01.02.04	Update
Controller	????-??	???.??.??	-	Update

20.8%

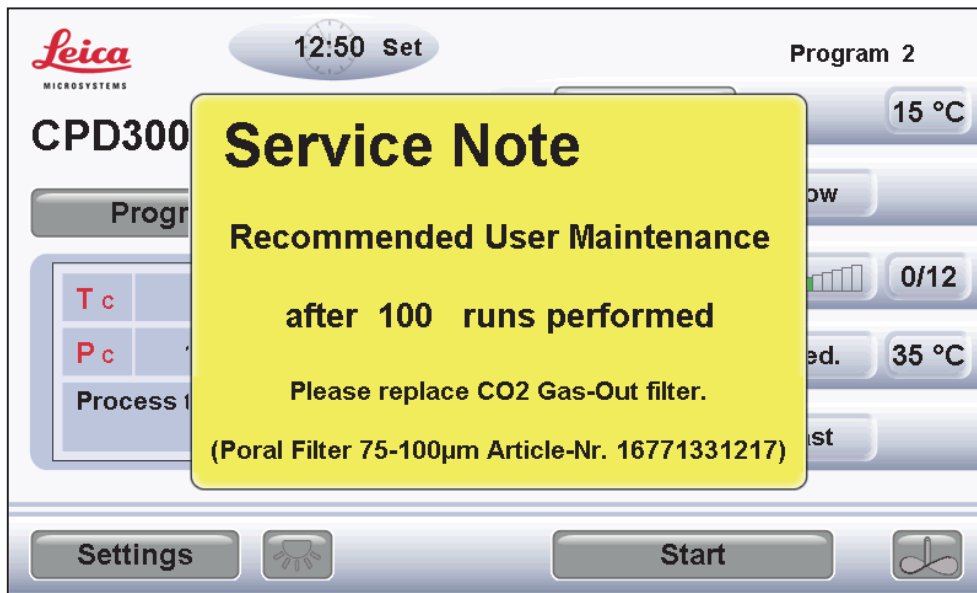
Back

15. Service Notes

15.1 Gas-Out filter maintenance



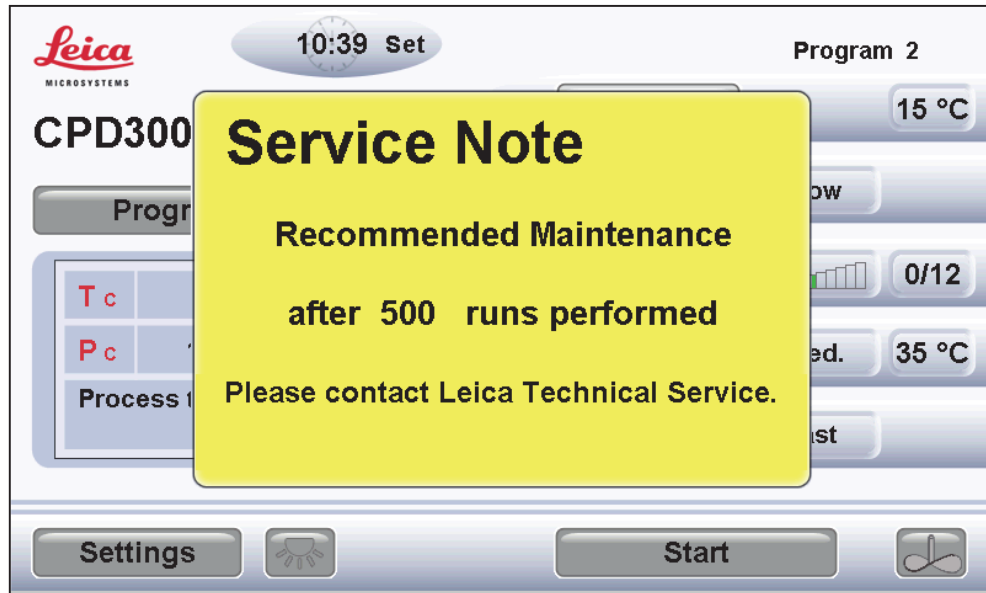
After 100 runs are performed, the following note appears:



Under Chapter 14 (Maintenance) the poral filter replacement is described.

15.2 General maintenance

After 500 runs a general maintenance is recommended!



16. User Maintenance



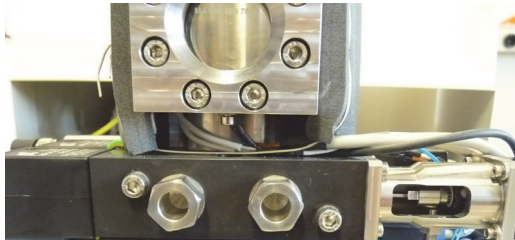
Before starting the maintenance, the pressure has to be released in the sample chamber, the CO₂-bottle has to be closed, and the CO₂ connection to the EM CPD300 has to be disconnected.

16.1 Poral filter replacement

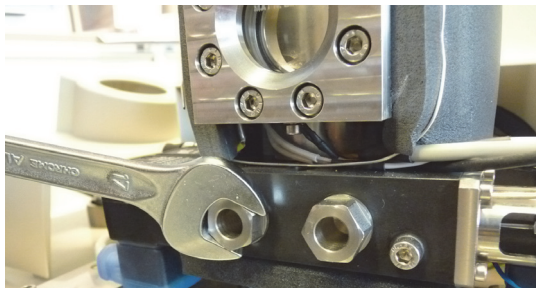
Remove front cover



Use a 17 size wrench to open the nut in the valve block.



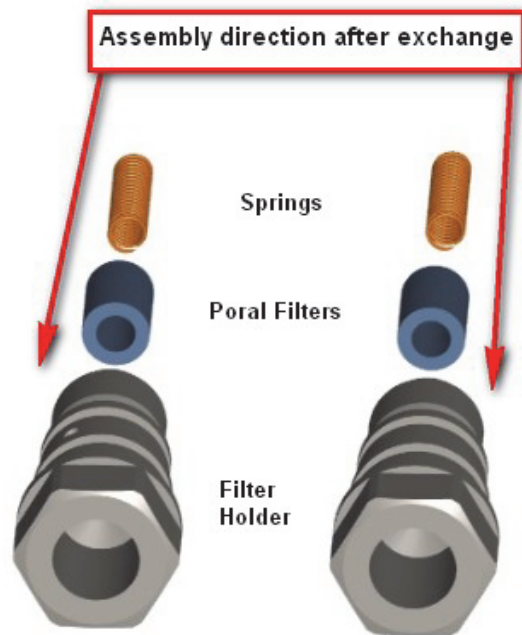
Remove nut, which includes porous filter, from the valve block.



Remove poral filter with spring from the nut.



When exchanging the poral filters the filters must be inserted into the filter holder so that the hole in the poral filter is pointing to the filter holder (the flat surface of the poral filter must point to the springs)



Exchange poral filter with the correct size (Poral filter (0.5 μm) for CO₂ Inlet (left) and Poral filter (75 - 100 μm) for CO₂ Outlet (right).
Reassemble poral filter in to the nut and screw nut in valve block.

16.2 Cleaning

All surfaces can be cleaned with aqueous reagents or 60% ethanol and a clean cloth.

17. Warnings and Troubleshooting

The list below summarizes Warnings (W...).

Regarding most of the displayed information, just follow the instructions; in the case of error messages, contacting your local Leica Service is required.

Code	Warning	Cause	Action
10E11	Com connection default		Call Leica service
10E12	Lid open	Process can not start if lid open	Close lid
10E13	Separator missing	Process can not start or continue if separator is missing	Insert separator
10E14	Separator full	Process can not start or continue if separator is full	Empty separator
10E16	Heatup error	Pressure too low	Check CO ₂ -bottle
10E17	Timeout CO ₂ -IN	CO ₂ -bottle empty or poral inlet filter is clogged	Check CO ₂ -bottle or exchange poral inlet filter
100E3	Pressure sensor failure		Call Leica service
100E4	Pressure fault	Poral outlet filter blocked	Exchange poral outlet filter
100E5	Temperature sensor failure		Call Leica service

Leica Microsystems CMS GmbH | Ernst-Leitz-Strasse 17-37 | D-35578 Wetzlar (Germany)
T +49 (0) 6441 29-0 | F +49 (0) 6441 29-2599

www.leica-microsystems.com

CONNECT
WITH US!

