Biolin Scientific
[Progress Together]

Attension High Pressure Chamber

User Manual

Original Instructions

MANUAL23748-4

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- EXPLOSION WARNING -

High Pressure Chamber is not explosion proof.

Attension High Pressure Chamber should only be used with chemicals and chemical combinations that are known to be safe to use at high pressures and temperatures. Using flammable fluids, or combination of fluids that can lead to potentially explosive reactions, is prohibited.

USE OF HIGH PRESSURE CHAMBER IN THESE POTENTIALLY HAZARDOUS APPLICATIONS IS PROHIBITED.

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Table of Contents

1 Basic information

High Pressure Chamber is made by Biolin Scientific and is to be used with Theta Flex or Theta Optical Tensiometer. High pressure chamber can be utilized in wide variety of applications where information on the high pressure/high temperature performance is required.

1.1 Notes on the Operating instructions

To prevent accidents, it is necessary to understand English to be able to read this manual. Otherwise, it is mandatory to receive complete instructions from someone qualified who understands both the instrument and English very well.

The present operating instructions describe the configuration of the system and provide information relating to its appropriate operation and maintenance. Please, thoroughly study the Operating instructions prior the first use of the High Pressure Chamber.

The Operating instructions include the safety notes, technical description, and instructions for usage as well as maintenance of the system. Please make sure to comply with all the operation and maintenance instructions provided in this manual to ensure full function and safety. The system may only be used for applications and purposes listed in these operating instructions. The manufacturer will not recognize claims due to inappropriate operation or insufficient maintenance.

All relevant accident prevention regulations and other generally accepted safety requirements must be observed and complied with.

1.2 Intended use

The High Pressure Chamber is to be used together with Theta Flex optical tensiometer. High Pressure Chamber can be used up to 400 bars and 200 ℃.

2 Safety notes

Installation and training is included in the High Pressure Chamber system and the system should never be used before the first training provided by the manufacturer. System should be installed against the wall to minimize the amount of personnel around the instrument. System is delivered with the safety cabinet that should be used at all times. Below, the requirements for operators as well as operation of the safety switches are explained.

The system must not be used before the manufacturer has installed the system and trained the users.

2.1 Requirements for the operator

All the operators need to receive an appropriate training from the manufacturer to operate the system. Each operator must have read and completely understood these operating instructions. The operator must understand that some fluids or fluid mixture might be dangerous to use at elevated pressures and temperatures. Using these fluids in the system needs to be carefully considered.

2.2 Safety switches

System contains safety switches for over pressure and overheating. The system contains two overpressure valves, one for the back of the chamber and the other for the front (see Fig. 1). The overpressure valves are set to about 410 bars. In case of over pressure, the pressure will be released through the overpressure valves. The valve panel needs to be closed during operation as the fluids that erupt from the chamber can be hot. After the overpressure incident, always evaluate what led to the overpressure to prevent it in the future.

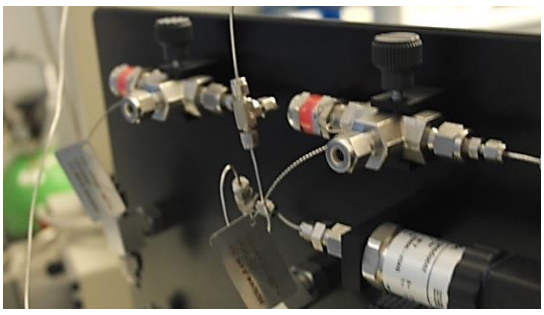


Fig. 1 Over pressure valves for back and front parts of the chamber

There are two levels of overheating switches in the system. The first one is embedded in the software. The software will prevent heating the chamber over the set temperature (set by manufacturer to 210 ℃). In case of software failure, the overheating switch is located at the top of the chamber (see Fig. 2) and is set to 225 ℃. After the over temperature incident, always evaluate what lead to the overheating to prevent it in the future.

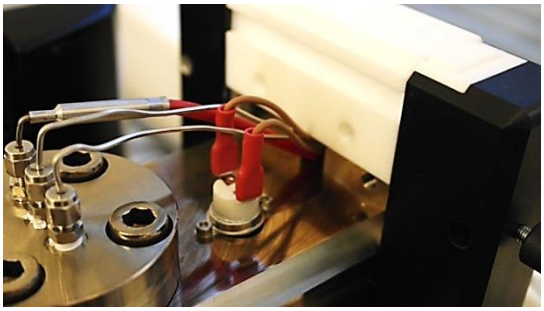


Fig. 2 Over temperature switch is located on top of the chamber

2.3 General safety precautions

WARNING!
The safety requirements listed in this manual must be followed in order to avoid personal injury and damage to the instrument. If the equipment is used in a manner not listed in this manual, protection provided by the equipment may be impaired.

WARNING!
RISK OF ELECTRICAL SHOCK. Do not connect this instrument to electrical power if the enclosure is damaged or any of the covers or panels are removed. Make sure the voltage rating on the instrument matches the line voltage available. Make sure the power cord is not damaged and it is properly connected to the instrument and a power outlet with protective earthing. Make sure that the power cord is easily accessible after the equipment has been installed and set at its working position.

WARNING!
RISK OF ELECTRICAL SHOCK OR FIRE HAZARD. The instrument has been designed for indoor use only. Do not expose it to rain, snow or dust. During storage or transport the instrument should be kept dry. Temperatures below 0°C and above 70°C should be avoided. Do not operate at ambient temperatures below 15°C and above 30°C.

WARNING!
RISK OF INJURY. Do not configure the instrument with parts that are not supplied by Biolin Scientific and not intended to be used with Attension instruments. Do not install substitute parts that are not described in this manual. Do not perform any modifications to the product.

If dangerous liquids are used, adequate protection such as proper ventilation, safety glasses, etc., should be used: refer to the safety information from the supplier and general safety regulations in your country. Carry out appropriate decontamination if equipment is exposed to hazardous material.

WARNING!
RISK OF BURNS. Exercise caution when heating the chamber. The chamber holder will reach dangerous temperatures when heated. The chamber holder is marked with hot surface warning symbol.

CAUTION!
Make sure that the power is switched off when making any electrical connections (apart from the USB cables). Connecting cables with power on may damage instrument electronics.

3 Technical description

3.1 Chamber parts

The cross-section of the chamber is shown in Fig. 3. The system consists of the stainless steel (EN 1.4401) chamber that enables surface and interfacial tension and contact angle measurements at high pressures and elevated temperatures. Three sapphire windows allow the visual view inside the chamber. O-rings are used as a seal. Viton O-rings come with the standard system but the O-ring material should be selected according to used fluids. Please, refer to material compatibility charts for appropriate O-ring materials for your applications. At the adapter flange on top of the chamber there are three connection ports for temperature probe and sample and bulk fluid introduction. As a unique feature, there is a possibility to use a piston inside the chamber which allows you to increase the pressure at the measurement compartment without changing the amount of bulk fluid (see more information on section 3.1.1). There are two connection ports located at the rear flange. This can be used to introduce the fluid at the rear compartment when piston is used. Chamber can be placed also upside down into the holder which allows the rising drop and captive bubble measurements.

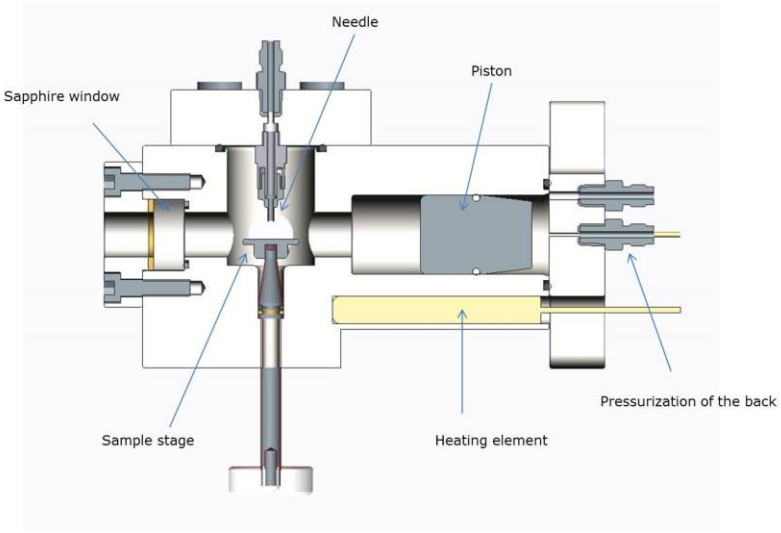


Fig. 3 Schematic of the chamber

Instrument is placed inside the safety cabinet which contains the needle valves used to control pressure inside the chamber. Safety cabinet can be open during the system set-up but should be closed whenever the chamber is pressurized or heated. The door containing the needle valves should be closed during standard operation but can be opened if maintenance of the valves needs to be done.

3.1.1 Piston

When working with liquids, it is possible to use the piston. There are several benefits that support the use of piston. Piston separates chamber into measurement compartment (front of the chamber) and pressurization compartment (back of the chamber). In case you are working with surfactants that are preloaded into chamber it is not possible to add any fluid into the chamber without changing the concentration. Using the piston, makes it possible to increase the pressure inside measurement compartment without adding any new fluid into it. Another benefit comes when working with harsh chemicals (e.g. highly concentrated brine) inside the chamber. By using the piston, it is possible to limit the chemicals almost completely to the measurement side keeping

the pump and most of the tubes only filled with water. Also, the contamination of the bulk phase is easier to minimize as liquid can be introduced into measurement part by using e.g. disposable syringe and nothing is added through the tube.

3.2 Assembly of the chamber

CAUTION! Chamber parts are heavy. Dropping a part may lead to personal injury or property damage.

Assembly of the chamber is started from the windows.

1. First the 20 x 2 O-ring is inserted.
2. Mount the sapphire window.
3. Place the brass sleeve on top of the window.
4. The window flange is attached by four M6 x 25 screws. Tighten the screws with the torque key to **10 Nm**.

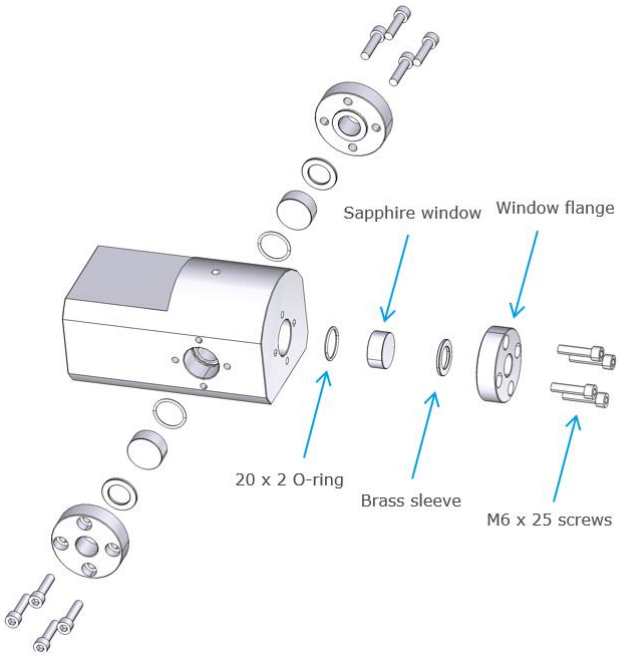


Fig. 4 Window assembly

All three windows are mounted the same way.

CAUTION! If there is any scratches or defect in the window, the window needs to be changed immediately. Failing to do so, may lead to serious accident.

The next assembly step is the rear flange. The flange is fixed by using four bolts. Appropriate O-ring is used as a seal. The assembly of the rear flange is shown in Fig. 5.

1. Insert the 34.59 x 2.62 O-ring.
2. If the piston is used, it needs to be inserted into the chamber before the rear flange is placed. Piston needs to be mounted with the 24 x 2.5 O-ring for proper seal. The piston can be inserted using the chamber holder tool included in the package. Insert the piston and press with the tool until the piston is completely inside the chamber.
3. If resistor heating is used, the heating bars need to be inserted before the rear flange is mounted. See more information about heating the chamber from section 4.2.
4. Tighten the flange by using four M10 x 40 screws to **47 Nm** with a torque key. You can use the tool provided to help to keep the chamber in place while tightening the screws.
5. Rear flange contains two inlets for fluids. The NPT male fluid inlets are connected by using Teflon tape and spanner to tighten. Teflon tape is rotated two rounds around the connector in the direction of the thread (see more information from 5.1.1). If the inlets are not needed (as in the case when piston is not used), the connectors can be replaced with the caps. Teflon tape is used in the same way as with the connectors. There is no need to remove the connectors after every measurement. Connectors should be replaced if proper cleaning of the chamber is done or they start to leak.

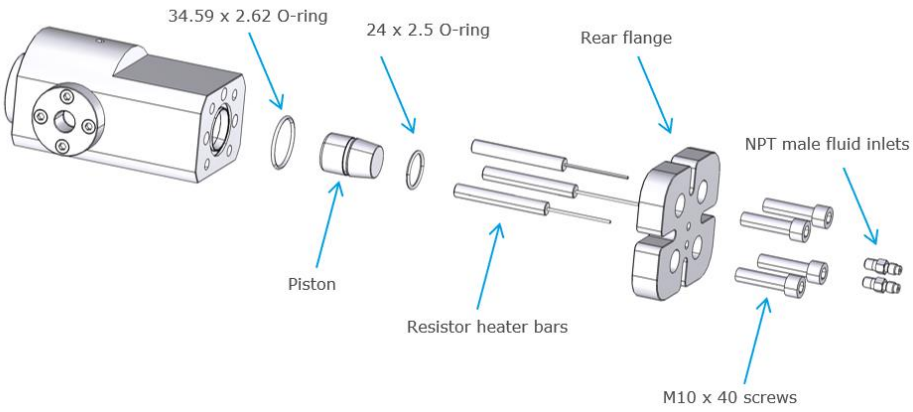


Fig. 5 Rear flange assembly

Next, mount the sample stage to the chamber. The mounting is shown in Fig. 6.

1. Insert 6.1 x 1.6 O-ring to the slot on the rod.
2. Mount sample stage to the rod with M3 x 4 screw.
3. Screw the rod to the chamber.
4. Mount the handle and the knob to the rod (Fig. 7).

The chamber delivery also includes sample holders that are screwed to the sample stage when used.

NOTE that if only surface / interfacial tension measurements are done, you should not place the sample stage but only insert the rod with the O-ring to seal the hole at the bottom of the chamber. In interfacial tension measurements several drops are usually used and they will accumulate on the sample stage which might interfere with the measurements.

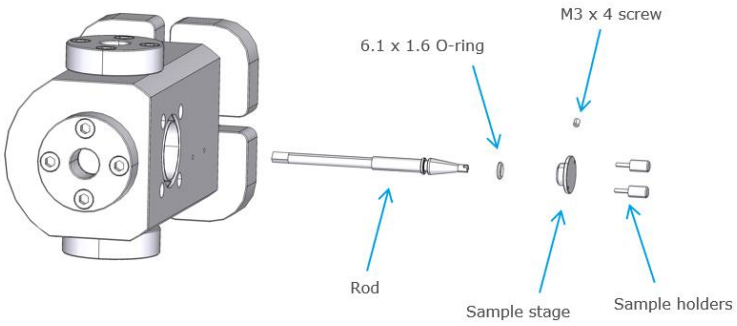


Fig. 6 Mounting the sample stage

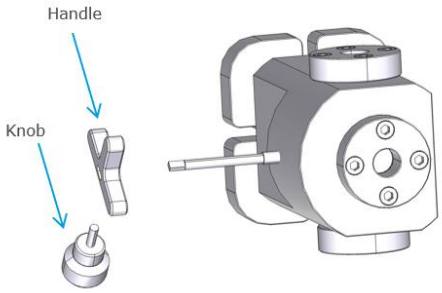


Fig. 7 Mounting the knob and handle

Next, the thermal safety switch is attached on top of the chamber.

1. Attach the thermal switch with two thumbscrews.
2. Attach the cables to the thermal switch as shown in Fig. 2. Note that if the cables are not connected, heating of the chamber is not possible.

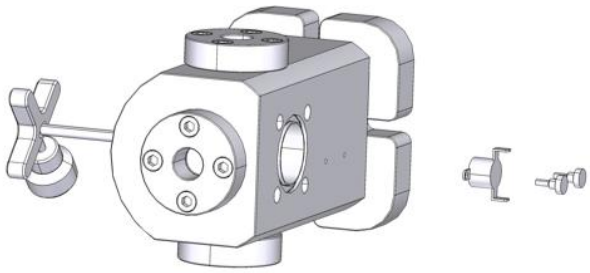


Fig. 8 Mounting the thermal switch

Before attaching the adapter flange on the top of the chamber, needle needs to be mounted. Needle is mounted as shown in Fig. 9.

1. Insert a 4.47 x 1.78 O-ring into male Luer lock
2. Mount male Luer lock to the adapter flange into the side that is inside the chamber.

3. Attach the needle to Luer lock.

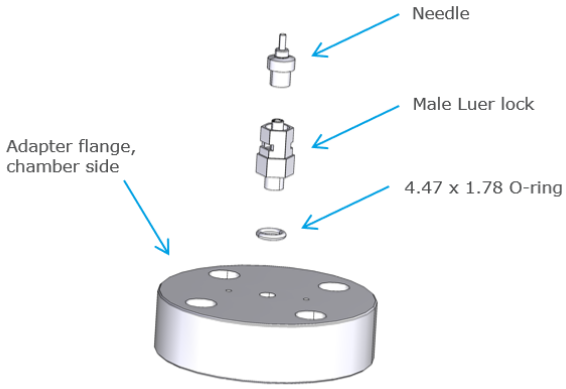


Fig. 9 Mounting the needle

There is no need to detach the needle after each measurement. When proper cleaning needs to be done, detaching the needle is advised.

The adapter flange is attached as shown in Fig. 10.

1. Insert 34.59 x 2.62 O-ring.
2. Flange is attached with four M10 x 40 screws. Tighten the four screws with the torque key to **47 Nm**.
3. Upper flange contains three inlets; one for the temperature probe, one for the needle and one for fluid introduction to the front of the chamber. Note that the connector for the temperature probe has little bit bigger hole in it to allow temperature probe to go through. The NTP male fluid inlets are connected by using Teflon tape and spanner to tighten. Teflon tape is rotated two rounds around the connector in the direction of the thread (see more information from 5.1.1). There is no need to remove the connectors after every measurement. Connectors should be replaced if proper cleaning of the chamber is done or they start to leak.

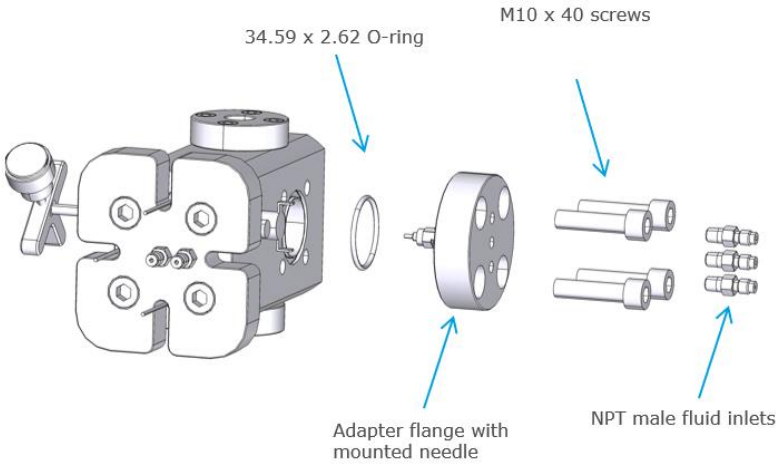


Fig. 10 Adapter flange assembly

CAUTION! If there is defect in the bolts or you noticed the bolt being jammed during disassembly, replace it immediately.

3.3 Valves and connections to the pumps

Two high pressure pumps are needed to operate the system. Depending on the fluids used, pumps can be either manual or automated. Most commonly an automated high pressure pump is used to introduce the fluid into the chamber. If gases are used, automated pump is mandatory, with liquids manual pump can be used as well. Sample introduction can be done with manual pump or automated pump.

In Fig. 11, the schematics of the High Pressure Chamber valves and fluid lines are presented. System is connected to two high pressure pumps of which the one is used to increase the pressure inside the chamber and the other one is for dispensing a fluid into chamber through the needle in the adapter flange. Pump symbols of the schematics in Fig. 11 will vary with different pump configurations, but the valve and fluid lines will remain the same. There are two connections for the pumps (bulk phase and dispenser inlets) on the front of the valve panel for easy connection of the pumps.

Fluid reservoir, that can be either gas or liquid, is connected to a pump generating the pressure inside the chamber. The pump is connected to bulk phase inlet, and from there the fluid line connects to both front and back valves, to allow the pressurization of the chamber either with or without piston. After the front valve, there is an overpressure valve to release the pressure from the front part of the chamber in case of overpressure situation. Front release valve is connected to same line, to allow the release of the pressure from the front part of the chamber. When piston is used, it is mandatory to connect the back release line since that is connected to overpressure valve for the back side of the chamber.

Pump for dispensing the fluid through the needle is connected to dispenser inlet. From there the fluid line connects through a needle valve to six-port valve shown in Fig. 12. The six-port valve has two positions as illustrated in Fig. 13. The first position enables injection of sample through the sample inlet to the needle, using a manual syringe. The first position also enables bleeding of carrier fluid line to a separate vessel. The second position connects the sample inlet to dispensing pump, and thus enables use of carrier fluid. Note that when operating the system, the six-port valve must be in position 2. **Never change the six-port valve position while the system is pressurized as this will lead to eruption of fluid from the chamber through the sample introduction port.**

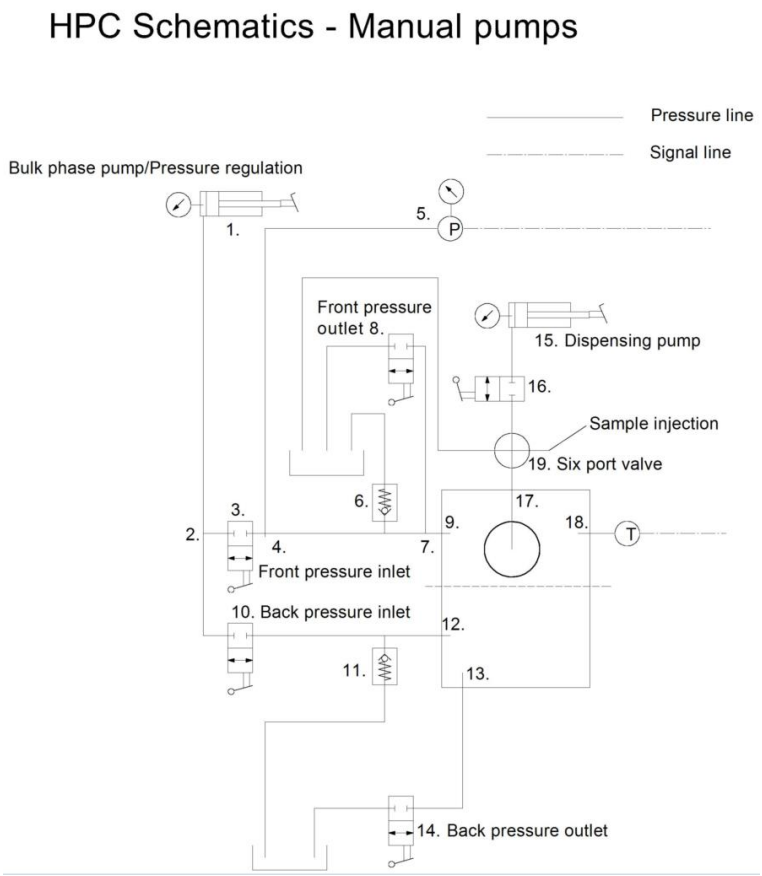


Fig. 11 Schematics of the valves and fluid lines into high pressure chamber when two manual pumps are connected. Please note that pump symbols of the schematics will vary with different pump configurations, but the valve and fluid lines will remain the same.

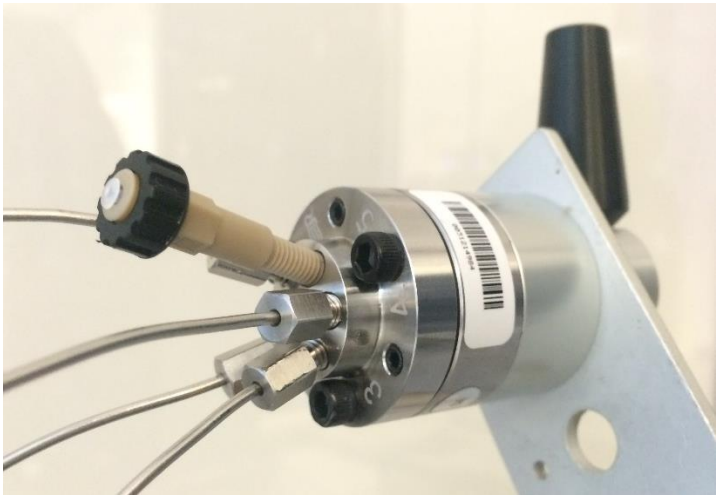


Fig. 12 Six-port valve.

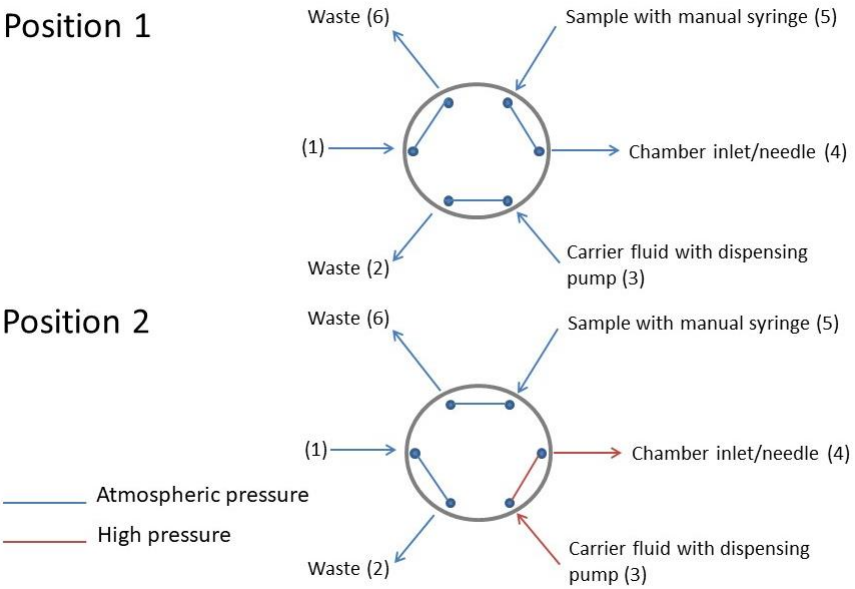


Fig. 13 Six port valve connections.

Needle valves are used in between the pumps and the chambers. The valves are shown in the Fig. 14.

Fig. 14 Valve panel on the safety cabinet door

All the tubes to the chamber go through the valve panel as shown in Fig. 15.

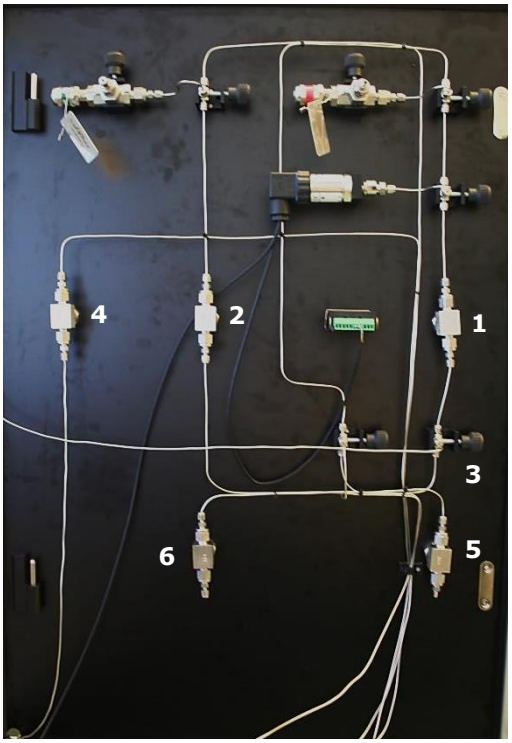


Fig. 15 Connection at the back of the valve panel; connection to front part of the chamber (1), connection to back part of the chamber (2), Connection to the pump for pressurizing front and back parts (3), Connection to sample pump and needle (4), Connections to release bottle for both front (5) and back (6) parts of the chamber.

The tubes are attached by rotating the cap on top of the NPT male connectors by hand. It should not take much force to rotate the tube in. When the connection is hand tight, tighten it further by rotating ¼ round clockwise as shown in Fig. 16. If new tubes need to be replaced and new connections made, please see the paragraph 5.1 for further instructions.

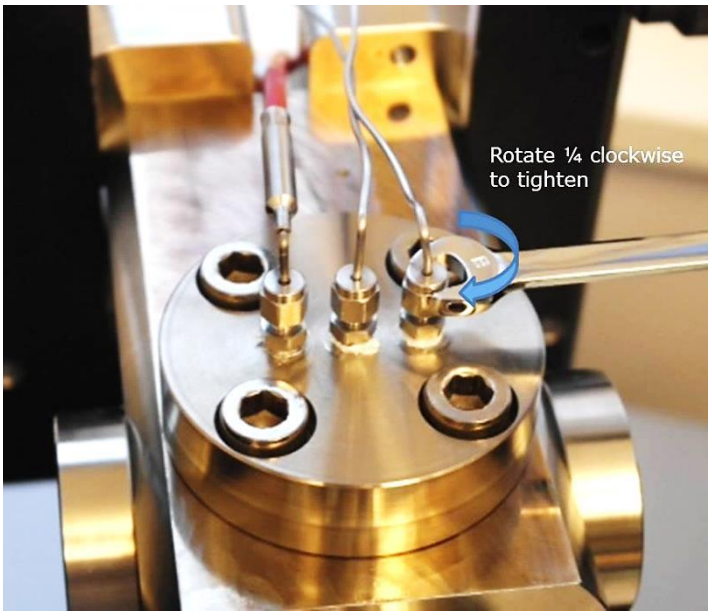


Fig. 16 Closing the tube connection

The tubes are color-coded depending on their use:

- Blue tube is the needle (the middle connection at the top flange)
- Red tube is the front pressure (the side connection at the top flange)
- White tube is the back inlet (on the back flange)
- Black tube is the back outlet (on the back flange)

3.4 Operation of pumps supplied by Biolin Scientific

Biolin Scientific offers the system with either manual or automated pumps. The selection of the pump depends on the fluids used in experiments as well as the desired automation level. For liquids, any pumps can be used for both introduction of the drop and pressurization of the chamber. When gases are used, the automated syringe pump is required.

3.4.1 Manual pump (62-6-10 Manual Pressure Pump Generators)

Manual pump needs to be filled manually with the liquid you want to use. It is advised to use only pure water inside the pump to prevent any contamination issues. If the pump is used to increase pressure inside the chamber, a piston should be utilized. If the pump is used to introduce the drop, then the sample is introduced through the six-port valve and water works only as a carrier fluid.

Manual pump can be filled by first removing the pressure gauge on top of the pump. Then the cylinder is filled by using syringe. The volume of the cylinder is about 30 ml.

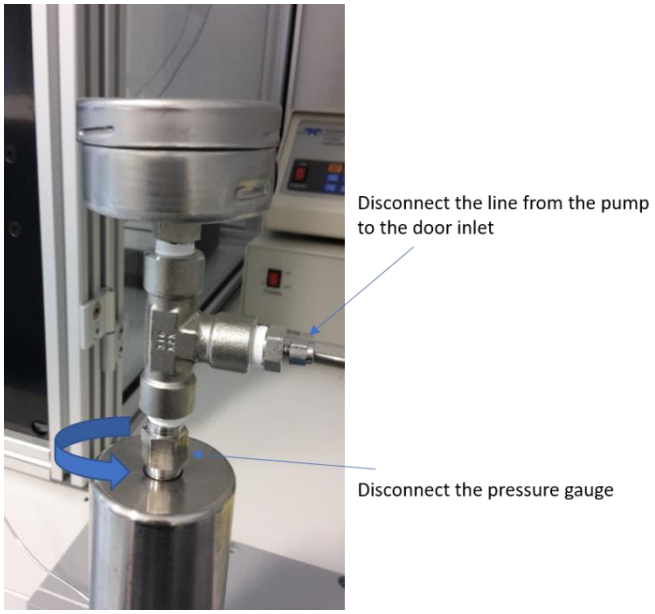


Fig. 17 Disconnecting the pressure gauge

Once the cylinder is filled the pressure gauge can be put back in place. It is good practice to drive some liquid through the tubes to bleed the system. The pressure is increased by simply turning the handle of the pump.

3.4.2 Automated pump (Teledyne ISCO 260D)

Here only the simple operation guidelines for automated pump are given. For more advanced use, please refer to pump manual.

It is advised to use only pure fluids inside the automated pump to prevent any contamination issues.

Description of the most used controls:

ZERO PRESS: Zeros the pressure of the pump. When the pump is depressurized, it is possible that the pressure reading deviates from zero due to pressure sensor drift. To zero the pressure, make sure that the port fittings are installed and the pump is depressurized, press ZERO PRESS. The display will show the current pressure and ask if you want to zero the pressure. Press A (as normally only one automated pump is attached and named pump A).

REFILL: To fill the pump, the pump needs to be connected to either gas bottle or the inlet tube should be immersed in the liquid. Press REFILL and open the gas bottle valves if gas is used. If filled with liquid, make sure to keep the inlet line immersed in the liquid few extra minutes after the cylinder is full as it takes some time for liquid to flow inside the cylinder.

CONST PRESS: To drive the pump, most typically constant pressure mode is used. This enables you to insert the desired pressure to the pump and the pump will automatically drive the cylinder as long as the set pressure is reached.

CONST FLOW: It is also possible to set the flow rate of the pump and drive with constant flow rate

LIMITS: The controller allows the user to set the minimum and maximum flow rate limits, the minimum and maximum pressure limits, and the maximum rate the pump will run while controlling the pressure in constant pressure mode.

RUN: When run is pressed, the pump will either drive the pump to set pressure (constant pressure mode) or with the set flow rate (constant flow rate mode)

STOP: Stops the pump

Fig. 18 Automated pump controller keypad

3.4.3 Automatic HPLC pump (Shimadzu LC-40D)

Here only simple operation guidelines for automatic HPLC pump are given. For more advanced use, please refer to pump manual.

It is advised to use only pure fluids inside the automatic HPLC pump to prevent any contamination issues.

Installation and initial pump settings

There are two red shipping screws on the bottom of the instrument. The screws must be removed prior installation. If the pump is later moved, the shipping screws must be used to prevent damaging the pump.

Turn on the pump at the back of the instrument and by pressing the power button on the lower left corner on the screen.

The liquid connections and drain valve knob are available under the front cover as shown in Fig. 19. The drain valve knob is opened only when the system is purged, otherwise it must be closed. Pump outlet connection, with metal tubing connected to it, is located under the drain valve knob. Pump inlet connection is located below the outlet connection. Drain tubing connection port is located on the right side of the drain valve knob.

Fig. 19 Automatic HPLC pump controller keypad and parts inside front cover

Change the display settings

As default, the display will turn off after 1 min of inactivity.

1) To increase the time, press → twice to see the auxiliary functions.
2) Select UTILITY by scrolling down with ↓ button. To enter the UTILITY press →.
3) Scroll down again by using ↓ button and press enter when text DISP OFF TIME is on the screen.
4) Set the time between 0 - 10 min. 0 (min) means that the display will always be on. Confirm with Enter.
5) To go back to initial screen press CE button until the initial screen is displayed.

Change the confirmation question

As a default, the pump asks confirmation if you want to turn on or off the pump. That makes it difficult to operate the pump, so it is advised to turn the confirmation question off.

1) Press → twice to see the auxiliary functions.
2) Select UTILITY by scrolling down with ↓ button. To enter the UTILITY press →.
3) Scroll down again by using ↓ button and press enter when text DIRECT KEY MODE is on the screen.
4) Press 1 to have direct key option selected. Confirm with Enter.
5) To go back to initial screen press CE button until the initial screen is displayed.

Change the pressure unit

1) Press → twice to see the auxiliary functions.
2) Select SYSTEM by scrolling down with ↓ button. To enter the SYSTEM press →.
3) Scroll down again by using ↓ button and press enter when text PRESSURE UNIT is on the screen.
4) Press 0 for MPa, 1 kgf, 2 bar, 3 psi, followed by Enter.
5) To go back to initial screen press CE button until the initial screen is displayed.

Change the operation mode

There are two operation modes in the HPLC pump; pressure and flow mode. Typically, the pump is used in flow mode.

1) To change the mode press → twice to see the auxiliary functions.
2) Select CONTROL by scrolling down with ↓ button. To enter the CONTROL press →.
3) Scroll down again by using ↓ button and press enter when text MODE CHANGE is on the screen.
4) Press 0 to have constant flow mode. Confirm with Enter.
5) To go back to initial screen press CE button until the initial screen is displayed.

Set max pressure

When operating in constant flow mode, it is advised to set a max pressure to the pump above which the pump will not go. The max pressure can be set as follows:

1) Press → twice to see the auxiliary functions.
2) Select PARAMETER by scrolling down with ↓ button. To enter the PARAMETER press →.
3) Scroll down again by using ↓ button and press enter when text P.MAX is on the screen.
4) Set the max pressure with numeric keypad. As the maximum pressure limit for the Attension Theta high pressure is 400 bar, it is advised not to set the max pressure higher than that. Confirm with Enter.
5) To go back to initial screen press CE button until the initial screen is displayed.

Zero pressure

Sometimes the displayed pressure in the pump needs to be zeroed. To do that, open the drain valve as shown in Fig. 20, to make sure there is no pressure in the pump.

1) Press → twice to see the auxiliary functions.
2) Select CONTROL by scrolling down with ↓ button. To enter the CONTROL press →.
3) Scroll down again by using ↓ button and press enter when text ZERO ADJUST is on the screen. Press Enter.
4) To go back to initial screen press CE button until the initial screen is displayed.

Purging the HPLC pump

It is important not to have any air in the HPLC pump as that can prevent full operation of the pump. When the pump is set up and whenever there is a chance that air has entered the pump, the pump must be purged with driving liquid as follows:

1) Place the inlet tube (with filter) into the bottle with the liquid used inside the pump (typically water).
2) Place the drain line and the stainless steel tube from the pump to waste.
3) Open the drain valve 180 degrees counter clockwise (see Fig. 20)
4) Press purge. The pump will now purge all lines for 3 min to get rid off any possible air in the tubes.
5) When the purge is finished, close the drain valve.
6) Test the liquid is coming out from the tube by pressing pump.
7) Connect the tube to valve panel.

Fig. 20 Drain valve knob

Operation of the pump

For standard operation constant flow mode is used. The flow rate setting range is from 0.0001 to 10 ml/min. If the pump is used for initial filling of the chamber, the highest flow rate of 10 ml/min can be used. However, lower rates (below 5 ml/min) are recommended when the pump is used to increase the pressure inside the chamber. Note that the pressure sensor inside the pump lags at higher flow rates. If exact pressure is needed, flow rate should be relatively low (less than 1

ml/min). It is also good to note that pump is not able to withdraw liquid back and thus overshooting the pressure should be avoided. This is especially important when the pump is used for droplet introduction. Then very low rates (around 0.1 ml/min and less) are recommended to be able to precisely control the volume of the drop.

Setting the flow rate

When operating in a constant flow mode, press enter when in initial screen. Use numeric keypad to enter the flow rate.

Running the pump

The pump is run by pressing PUMP. The pump will now pump with the set flow rate. The pressure will start to increase on the pump display.

3.5 Disassembling the chamber

The chamber is disassembled by first disconnecting all the fluid lines and temperature probe from the chamber. Remove also the cables from the thermal switch. Lift the chamber carefully from the holder. Disassembly in reverse order than assembly was done. There is no need to detach the NPT fluid inlets unless leakage was detected or the chamber needs to be cleaned.

If the piston has been used, it can be pulled out by using the tool provided with the instrument.

CAUTION! Before disassembling the chamber make sure that it is depressurized. Opening the bolts before depressurization can lead to serious personal injury.

4 Operation

This chapter describes the operation of the High Pressure Chamber system and the software in the parts that are unique for the system. To learn more about using OneAttension software for contact angle and surface/interfacial tension measurements, see OneAttension Theta Flex user manual.

4.1 Calibration of the image

The image of Theta Flex needs to be calibrated to be able to define the size of one pixel in the image. This information is used to calculate surface and interfacial tensions and volumes of the droplets. The calibration is done by using the needle. Needle diameter is announced in gauges (G) which can be converted to mm. Table below shows the typical diameters of the needles provided by Biolin Scientific. However, it is always advised to measure the needle diameter as they may vary slightly from needle to needle.

Needle gauge	Diameter (mm)
14 G	2.108
22 G	0.718
30 G	0.311

Before calibration it is good to adjust the camera parameters to be optimum. This is most easily done by selecting camera parameters below the image and pressing the auto adjust button. Make sure that there is nothing in the image while doing that. Software will now automatically adjust the camera parameters to provide you with the best possible image. Then, bring the needle into the view. It does not matter if the needle comes from the bottom or on top of the image. Have a good portion of needle visible in the image as show in Fig. 21. Press calibrate button at the bottom of the image and choose "use needle calibration". Insert the proper needle diameter. If the chamber is placed upside down (needle is at the bottom of the image), select flip-Y as well. Click calibrate. The calibration is now done.

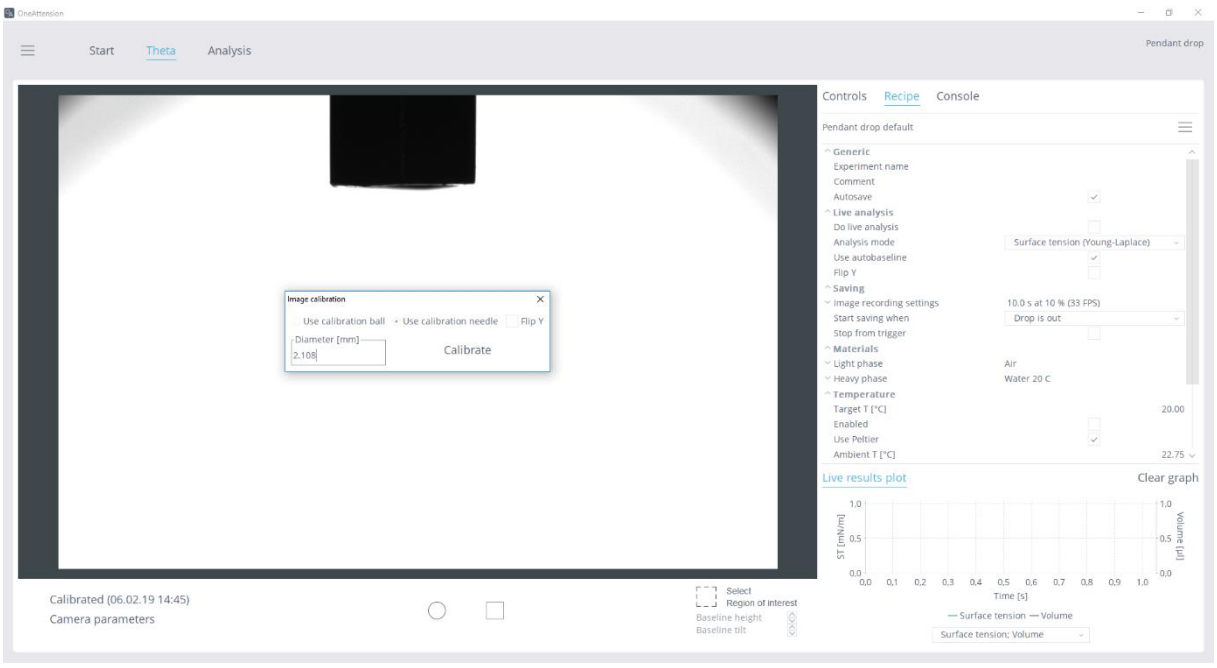


Fig. 21 Calibration of the image

If there is an error massage "Couldn't analyze image". Adjust the camera parameters and focus and try again.

After calibration is done, you should not change the zoom of the lens anymore as that affects the calibration factor. In case zoom is changed, please re-calibrate to obtain correct results.

4.2 Heating the chamber

The chamber can be heated by using either resistor heaters or by using the Peltier element. Peltier element can be used in the range of 1 to 70 °C and the working range for resistor heater is up to 200 °C. Controlling the temperature is recommended even if measurements at room temperature are done since that will significantly reduce the stabilization time for the pressure.

Note that for safety reasons, the heating should always be done prior increasing the pressure. This is because heating can cause a sudden increase in pressure.

It is always mandatory to use the insulation cover for safety reasons and when heating is used, the insulation cover will also significantly reduce the heat loses from the chamber.

The insulation cover is composed of three separate parts (see Fig. 22.); insulation frame, back part and bottom part.

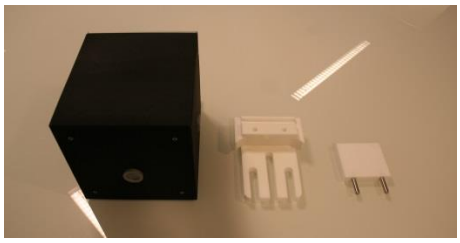


Fig. 22 Insulation cover parts from left to right; Insulation frame, back part and bottom part

If you are using the resistor heater, place the bottom part as shown in Fig. 23. If you are using the Peltier element, the bottom part is replaced by the Peltier element. In case the chamber is turned upside down the bottom part will come to the top.

Fig. 23 Bottom part of the insulation cover in place

Then insert the chamber into the holder. Place the back part in; make sure that all the cables fit in nicely. Slide in the insulation frame. Note that it is important to have the tubings to the chamber bend for insulation frame to fit as shown in Fig. 24.

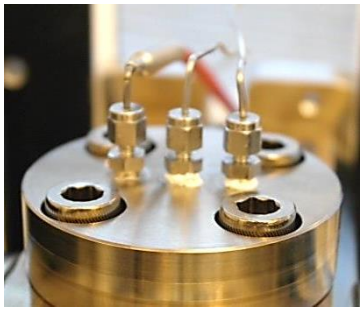


Fig. 24 Connections from the upper flan

4.2.1 Resistor heating

CAUTION! Chamber should always be heated first before pressurizing the chamber. Heating up fluids in closed chamber can lead to sudden increase of pressure.

Resistor heating can be used in the temperature range up to 200 °C. Before enabling the heating, make sure the resistor bars are correctly in place, the temperature switch is on top of the chamber and connected and the temperature probe is in place. Insert the insulation cover parts.

The heating is controlled through OneAttension software. Set the desired temperature as show in Fig. 25 . The heating is started when "enable" is selected. Also note that "Use Peltier" setting is selected as default, it must be deselected to start heating with resistors.

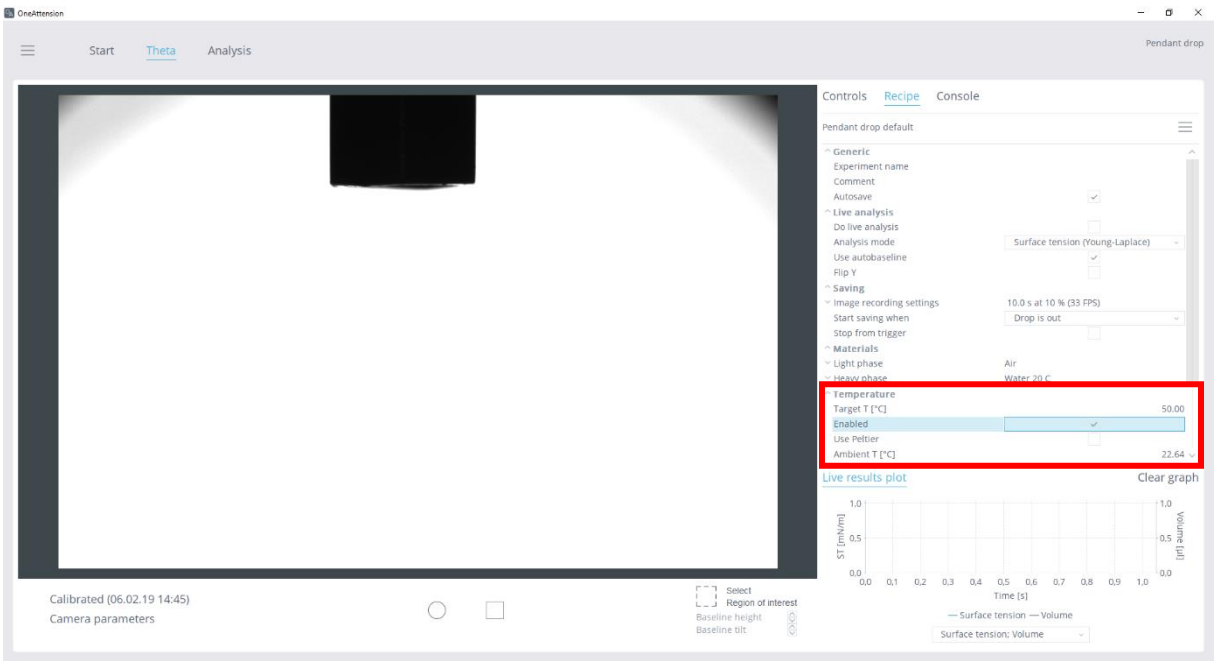


Fig. 25 Setting the temperature with resistor heaters

CAUTION! Make sure the temperature probe is inside the chamber before enabling the heater. Serious overheating can occur if temperature probe is not properly in place.

After desired temperature is reach, the pressurization of the chamber can be done. Note that letting fluids into the chamber may cause the temperature to decrease temporarily.

To turn off the heating, deselect enable. Note that there is no active cooling with the resistor heaters and it can thus take several hours for the temperature to decrease to room temperature. Before removing the insulation cover, make sure the temperature is decreased below 40 ℃.

CAUTION! Heated chamber can be extremely hot. Do not remove the insulation cover while the temperature is above 40 ˚C.

4.2.2 Peltier heating

Peltier element can be used for cooling and heating the chamber in a range between 1 to 70 ˚C. When Peltier element (see Fig. 26) is used, it replaces the bottom part of the insulation cover. Before starting the cooling/heating, make sure the temperature probe and the insulation cover are properly in place.

To enhance cooling capability of the Peltier element, Julabo water bath is connected. Both Peltier and Julabo temperature can be controlled through OneAttension software. For additional information about the Julabo settings and connection to a computer, please see a separate Julabo setup guide.

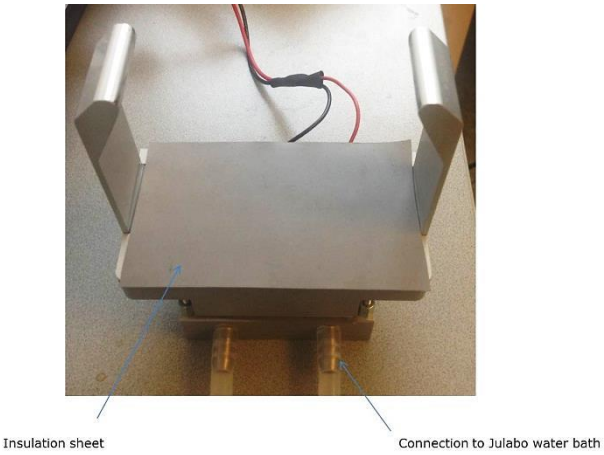


Fig. 26 Peltier element connected to Julabo water bath

When Peltier is used and Julabo water bath connected, the temperature control will appear in the recipe as shown in the Fig. 27. Target temperature is set under temperature controls and "use Peltier" is selected. There is a possibility to either let the software to control the Julabo temperature according to a target temperature or set the Julabo temperature manually. To let the software to do the control, select "Controller changes bath target". The Julabo temperature is now set to be the same as the target temperature. To control the temperature manually, set the desired temperature as bath target temperature. By setting the temperature manually it is possible to make the heating/cooling little bit faster by letting the Julabo pre heat/cool the Peltier element. To turn on the Peltier select enabled. To turn on the Julabo, select circulator on.

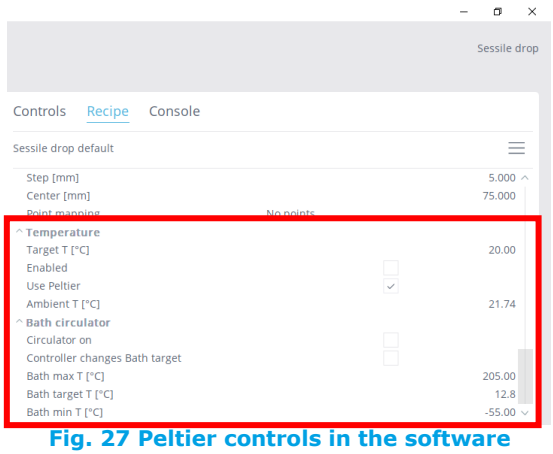


Fig. 27 Peltier controls in the software

4.3 Step-by-step instructions for measurements

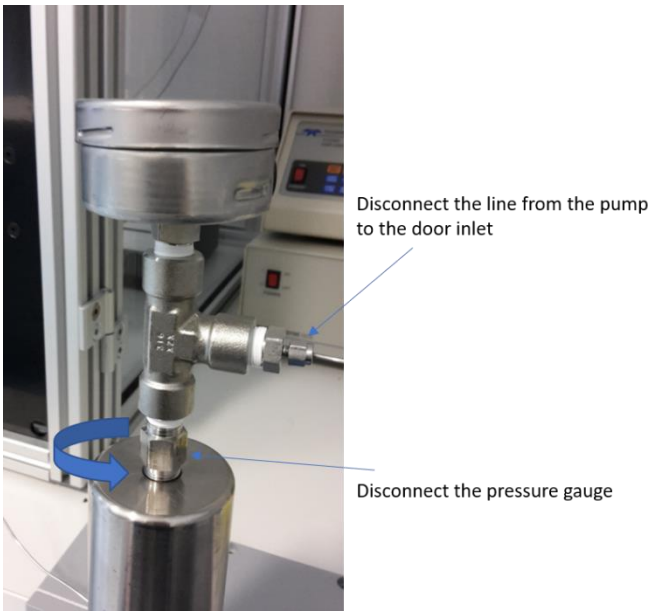
In this part of the manual, step-by-step instructions for the successful measurements are given. Preparation of manual and automated pump as well as filling the sample loop are first explained in detail. As the operation of the system is slightly different whether you are working with gas or liquid as a bulk phase, these two situations are handled separately. Apart from these two clear cases, it is possible to have situation where the fluid changes phase at certain temperature and pressure point, for example when working with CO_2. Special features related to CO_2 are also discussed.

CAUTION! Make sure you are always aware of the pressures at different parts of the system. There can be different pressures inside the chamber, the tubes and the pump.

Preping the manual pump

Before starting the measurements, it is important to make sure that there is no air in the manual pump or the tubes from the pump to the chamber. This is especially important when manual pump is used to introduce the drop.

1) Push any remaining liquid out of the pump, if necessary clean the pump as instructed in 4.4.4.
2) Disconnect the tube that goes from the manual pump to the cabinet door and remove the pressure gauge.



3) Drive the piston down by rotating the handle.
4) Fill the pump with the syringe. Most commonly water is used as a liquid inside the pump.
    a. If manual pump is used as a drop phase pump, the sample is introduced through the six-port valve (filling the sample loop)
    b. If manual pump is used as a bulk phase pump, the pump can be also filled with the bulk liquid. If high concentration salt solutions or other harsh liquids are used as bulk phase, it is advisable to use piston instead (see 4.3.3).
5) Attach pressure gauge and tube line back in place.
6) Rotate the handle and check that liquid is coming through the needle line (check six-port valve in both positions) /bulk phase line to make sure that there is no air in the system.

Preping the automated pump

1) Before filling the pump, clean the pump according to manufacturer recommendations if needed.
2) Make sure all the valves on a valve panel are closed.

3) Fill the automated pump. Note that it is recommended to use only pure fluids in the pump as cleaning of the pump can be tedious.
    a. With liquid; empty the cylinder and immerse the inlet line of the pump to the liquid and press refill. Make sure to keep the line immersed few minutes after the cylinder is full as it takes some time for the liquid to flow inside the pump.
    b. With gas/CO_2; Open the gas bottle valves and press refill.
4) Remember to close the valves to the gas bottle / liquid container after the cylinder is full.
5) Make sure all the tube lines are filled with the fluid.
    a. With liquid; open the front pressure inlet valve (or dispenser inlet valve) and press run to push some liquid through the lines.
    b. With gas/CO_2; open the front pressure inlet valve (or dispenser inlet valve) and let some gas go through the lines.

Preping the automatic HPLC pump

1) Place the inlet tube line in the bottle that contains liquid you want to use inside the pump. It is advised to use only water to avoid any contamination issues. Place the drain line in the waste as well as the outlet line (either needle line or front or back pressure line).
2) Make sure the valve where the pump is connected is open in the valve panel door
3) Open the drain valve
4) Press purge. The pump will now purge all the lines for 3 min to get rid of all the air in the tubes.
5) Close the drain valve and keep it close for rest of the measurements
6) Test that liquid is coming from the output tube by pressing the pump button

Filling the sample loop (only in case of liquid samples)

1) If the pumps have been prepped, the 6-port valve lines should now be filled with carrier fluid (usually water),
2) Check that the 6-port valve is in position 1.
3) Push the sample through the sample introduction inlet with the help of a syringe. Note that degassing the sample might be needed.
4) Sample loop is filled when you can see sample coming out of the needle line.
5) Change the valve position to 2. The valve position should not be changed anymore.

Using the eccentric needle

Eccentric needle can be used to create several sessile drops on the same sample. Droplets are placed on the edge of the sample, which allows a new droplet to be placed when the sample stage is rotated. When mounting the eccentric needle to the adapter flange, make sure the needle is positioned perpendicular to the orientation of the three inlets on top of the flange. This way the needle will be perpendicular to the camera view (Fig. 28) and there is more space to create droplets.

Place the first droplet normally on top of the sample. Then rotate the sample stage so that the first droplet is not underneath the needle. Keep the distance between the needle and the sample short so that the second droplet will almost touch the sample when it is created. Then place the second droplet on the sample. Use **ROI** (Region Of Interest) function to differentiate the droplets for the analysis.

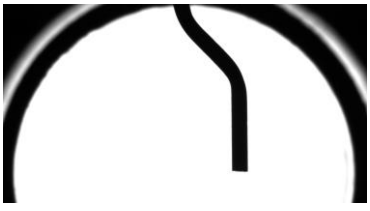


Fig. 28 Eccentric needle perpendicular to the camera view.

4.3.1 Using gas as a bulk phase

Final preparations of the system

1) Assemble the chamber as instructed in 3.2. As piston is not used, the rear flange inlets can be plugged.
2) Connect all the connection (temperature probe, needle line, bulk phase line) to the adapter flange.
3) Check that all the valves in the valve panel are closed.
4) If heating is used, double check that the over temperature switch is connected.
5) Put the heating insulation cover in place.
6) Check that the needle is visual in the image and calibrate (see 4.1).

Making the measurements

1) In case heating is used, heat the chamber first to desired temperature.
2) When gases are used, the pressure inside the pump is usually the same as the pressure inside the gas bottle. To get the pressure lower than that in the pump, the front pressure inlet valve needs to be opened carefully to let only small amount of the gas to enter the chamber.
3) Once the desired temperature and pressure are reached, the drop can be created. Increase the pressure of the drop phase pump as close to the pressure inside the chamber as possible.
4) Open the dispenser inlet valve slowly monitoring the needle at the same time.
5) When the valve is open and there is still no fluid coming out of the needle, increase the pressure in the drop phase pump slowly. Monitor the needle carefully at the same time.
6) When the drop is coming out, close the dispenser inlet valve. Note that there is some dead volume in the valve. To make the drop of desired size, you might need to take some of the sample back while closing the valve.
7) Once the drop is created, the recording can be done. For
    a. Pendant drop measurement, press record to record the pendant drop.
    b. Contact angle measurements, lift the sample stage to place the drop on the sample. Note that this must be done at relatively low pressure (about 50 bar). Press record.
8) To continue measurements, increase the pressure again by slowly opening the front pressure inlet valve or increasing the pressure with the pump. Make a drop and record. In contact angle measurements, same drop is used in all pressures.
9) When working with gases, it might be necessary to re-fill the pump cylinder. To do that, close all the valves in the valve panel. Open the gas bottle valves and press refill.
10) Continue the measurements by increasing the pressure in the pump to the pressure inside the chamber before opening the front pressure inlet valve.

End of measurements

1) When you are ready with your measurements, release the pressure inside the chamber by opening the front pressure outlet valve. Note that if very high temperatures have been used, you might want to let the chamber to cool down before releasing the pressure.
2) Before disassembling the chamber, it is advised to do some initial cleaning of the six-port valve, especially if samples like crude oil, have been used.
    a. Rinse the six -port valve in both positions (1 & 2) by using carrier fluid pump. If needed, you can even increase the pressure in the pump before opening the dispenser inlet valve to push all the excess sample out of the tubes.
    b. Rinse the valve also with appropriate solvent by pushing it through the sample introduction port with a syringe.
3) Disassemble the chamber by disconnecting all the tubes. Be careful in case of any liquid coming out while doing that.

4) When the system is shut down, you should first turn off the software, then Theta Flex and the electronic box.

4.3.2 Using liquid as a bulk phase (without piston)

Pre-filling the chamber

When piston is not used, the bulk liquid inside the chamber and the pump used to pressurize the chamber should be the same. It is important that the pump is also very clean as the liquid in the pump will enter the chamber. At least when manual pump is used to pressurize the chamber, it is advised to pre-fill the chamber by using syringe as the volume of the chamber (85 ml) is much bigger than the volume of the manual pump (30 ml).

1) Assembly the chamber as described in 3.2. As piston is not used, the rear flange inlets can be plugged.
2) Lift the chamber to the holder. Do not connect any lines yet.
3) Pre-fill the chamber by pushing liquid through one of the adapter flange inlets with a syringe. Keep other inlets open for air to come out. Pre-filling of the chamber can be done either by using a disposable syringe or with the help of automated HPLC pump (flow rate can be set to 10 ml/min)
4) Once there is liquid coming through one of the inlets, lift the chamber off the holder and turn side to side to get any trapped air out of the chamber.
5) Continue filling with the syringe until you see liquid coming out

Final preparations of the system

1) Connect all the connection (temperature probe, needle line, bulk phase line) to the adapter flange.
2) Check that all the valves in the valve panel are closed.
3) If heating is used, double check that the over temperature switch is connected.
4) If lighter phase is coming from the needle, the chamber needs to be turned upside down at this point. Be careful when doing that as the chamber is very heavy.
5) Put the heating insulation cover in place.
6) Check that the needle is visual in the image and calibrate.

Making the measurements

1) In case heating is used, heat the chamber first to desired temperature.
2) Open the front pressure inlet valve. To get to desired pressure either
    a. Set the desired pressure to the automated pump, press run.
    b. Set the flow rate on your HPLC pump (e.g. 1 ml/min) and press pump. Press pump again when desired pressure is reached.
    c. Increase the pressure of the manual pump by rotating the handle.
3) Once the desired temperature and pressure are reached, the drop can be created. Increase the pressure of the drop phase pump as close to the pressure inside the chamber as possible.
4) Open the dispenser inlet valve slowly monitoring the needle at the same time.
5) When the valve is open and there is still no fluid coming out of the needle, increase the pressure in the drop phase pump slowly. Monitor the needle carefully at the same time.
6) When the drop is coming out, close the dispenser inlet valve. Note that there is some dead volume in the valve. To make the drop of desired size, it is advised to start with the smaller drop and close the valve almost completely before finetuning the size of the drop.
7) Once the drop is created, the recording can be done. For
    a. Pendant drop measurement, press record to record the pendant drop.

b. Contact angle measurements, lift the sample stage to place the drop on the sample. Note that this must be done at relatively low pressure (about 50 bar). Press record.

8) To continue measurements, increase the pressure again, make a drop and record. In contact angle measurements, same drop is used in all pressures.

End of measurements

1) When you are ready with your measurements, release the pressure inside the chamber by opening the front pressure outlet valve. Note that if very high temperatures have been used, you should let the chamber to cool down before releasing the pressure.
2) Before disassembling the chamber, it is advised to do some initial cleaning of the six-port valve, especially if samples like crude oil, have been used.
   a. Rinse the six -port valve in both positions (1 & 2) by using carrier fluid pump. If needed, you can even increase the pressure in the pump before opening the dispenser inlet valve to push all the excess sample out of the tubes.
   b. Rinse the valve also with appropriate solvent by pushing it through the sample introduction port with a syringe.
3) Disassemble the chamber by disconnecting all the tubes. Be careful in case of any liquid coming out while doing that.
4) When the system is shut down, you should first turn off the software, then Theta Flex and the electronic box.

4.3.3 Using liquid as a bulk phase (with piston)

Pre-filling the chamber

When using piston, it is possible to increase the pressure without adding anything to the measurement part of the chamber. This makes it possible to use relatively harsh bulk phase (e.g. concentrated brine) and minimize the contact between the bulk phase and the parts of the system (e.g. most of the tubes and pump). Also, contamination of the bulk phase is much easier to control as the chamber can be filled with bulk phase liquid by using disposable syringe.

6) Assembly the chamber as described in 3.2. As piston is used, the rear flange inlets must be open.
7) Lift the chamber to the holder. Do not connect any lines yet.
8) Pre-fill the chamber by pushing liquid through one of the adapter flange inlets with a syringe. Keep other inlets open for air to come out. Pre-filling of the chamber can be done either with the disposable syringe or with the help of automated HPLC pump (flow rate can be set to 10 ml/min).
9) Once there is liquid coming through one of the inlets, lift the chamber off the holder and turn side to side to get any trapped air out of the chamber.
10) Continue filling with the syringe until you see liquid coming out

Final preparations of the system

1) Connect all the connection (temperature probe, dispenser line, front pressure inlet line) to the adapter flange. Note that it is important to connect also the front pressure inlet line as this is the line where the pressure inside the measurement compartment is measured. Connect also the back pressure inlet and outlet lines.
2) Check that all the valves in the valve panel are closed.
3) If heating is used, double check that the over temperature switch is connected.
4) If lighter phase is coming from the needle, the chamber needs to be turned upside down at this point. Be careful when doing that as the chamber is very heavy.
5) Put the heating insulation cover in place.
6) Check that the needle is visual in the image and calibrate.

Making the measurements

1) In case heating is used, heat the chamber first to desired temperature.
2) Open the back-pressure inlet valve. To get to desired pressure either
    a. Set the desired pressure to the automated pump, press run.
    b. Set the flow rate on your HPLC pump (e.g. 1 ml/min) and press pump. Press pump again when desired pressure is reached.
    c. Increase the pressure with the manual pump by rotating the handle

    The piston inside the chamber will now move and the pressure in the measurement compartment increases. Note that there is a slight pressure drop over the piston and thus the pressure at the back of the chamber is slightly higher than that of the measurement compartment.
3) Once the desired temperature and pressure are reached, the drop can be created. Increase the pressure of the drop phase pump as close to the pressure inside the chamber as possible.
4) Open the dispenser inlet valve slowly monitoring the needle at the same time.
5) When the valve is open and there is still no fluid coming out of the needle, increase the pressure in the drop phase pump slowly. Monitor the needle carefully at the same time.
6) When the drop is coming out, close the dispenser inlet valve. Note that there is some dead volume in the valve. To make the drop of desired size, it is advised to start with a smaller drop and close the valve almost completely before finetuning the size of the drop.
7) Once the drop is created, the recording can be done. For
    a. Pendant drop measurement, press record to record the pendant drop.
    b. Contact angle measurements, lift the sample stage to place the drop on the sample. Note that this must be done at relatively low pressure (about 50 bar). Press record.
8) To continue measurements, increase the pressure again, make a drop and record. In contact angle measurements, same drop is used in all pressures.

End of measurements

1) When you are ready with your measurements, release the pressure inside the chamber by opening the back pressure outlet valve. Note that if very high temperatures have been used, you should let the chamber to cool down before releasing the pressure. Release the pressure from the front as well by opening the front pressure outlet valve.
2) Before disassembling the chamber, it is advised to do some initial cleaning of the 6-port valve, especially if samples like crude oil, have been used.
    a. Rinse the 6 -port valve in both positions (1 & 2) by using carrier fluid pump. If needed, you can even increase the pressure in the pump before opening the dispenser inlet valve to push all the excess sample out of the tubes.
    b. Rinse the valve also with appropriate solvent by pushing it through the sample introduction port with a syringe.
3) Disassemble the chamber by disconnecting all the tubes. Be careful in case of any liquid coming out while doing that.
4) When the system is shut down, you should first turn off the software, then Theta Flex and the electronic box.

4.3.4 Using CO_2 as the bulk phase (with piston)

Using CO_2 as the bulk phase has features from both operations with gases and liquids. This is because the phase of CO_2 depends on the used temperature and pressure combination. Unlike with typical gases, the use of piston with CO_2 is possible when operating in supercritical or liquid phases. As the operation of the instruments is exactly the same with normal gases as when pressurizing with CO2 without piston, this is not separately explained here. The operation with piston is explained more in detailed.

Final preparations of the system

1) Connect all the connection (temperature probe, needle line, bulk phase line) to the adapter flange. Note that it is important to connect also the front pressure inlet line as this is the line where the pressure inside the measurement compartment is measured. Connect also the back pressure inlet and outlet lines.
2) Check that all the valves in the valve panel are closed.
3) Attach the over temperature switch connectors.
4) Put the heating insulation cover in place.
5) Check that the needle is visual in the image and calibrate.

Making the measurements

1) You might want to first purge the chamber with CO_2 to get rid of excess air in the chamber. This is done by opening the front pressure inlet valve and letting some CO_2 to enter the chamber. Close the front pressure inlet valve and open the front pressure release valve.
2) In case heating is used, heat the chamber first to desired temperature.
3) Most typically the pressure in the CO_2 tank is around 55 bars depending on the temperature. Fill the front part of the chamber with CO_2 by opening the front inlet valve. Note that you need to increase the pressure by using the front inlet until you reach either a supercritical or liquid state. Only after that the piston can be utilized.
4) To use the piston, open the back-pressure inlet valve and set the desired pressure into automated pump and press run. The piston inside the chamber will now move and the pressure in the measurement compartment increases. Note that there is a slight pressure drop over the piston and thus the pressure at the back of the chamber is slightly higher than that of the measurement compartment.
5) Once the desired temperature and pressure are reached, the drop can be created. Increase the pressure of the drop phase pump as close to the pressure inside the chamber as possible.
6) Open the dispenser inlet valve slowly monitoring the needle at the same time.
7) When the valve is open and there is still no fluid coming out of the needle, increase the pressure in the drop phase pump slowly. Monitor the needle carefully at the same time.
8) When the drop is coming out, close the dispenser inlet valve. Note that there is some dead volume in the valve. To make the drop of desired size, it is advised to start with a smaller drop and close the valve almost completely before fine tuning the size of the drop.
9) Once the drop is created, the recording can be done. For
    a. Pendant drop measurement, press record to record the pendant drop.
    b. Contact angle measurements, lift the sample stage to place the drop on the sample. Note that this must be done at relatively low pressure (about 50 bar). Press record.
10) To continue measurements, increase the pressure again by opening the back pressure inlet valve and set the desired pressure into automated pump. Press RUN. Make a drop and record. In contact angle measurements, same drop is used in all pressures.
11) When working with CO_2, it might be necessary to re-fill the pump cylinder. To do that, close all the valves in the valve panel. Open the gas bottle valves and press refill.
12) Continue the measurements by increasing the pressure in the pump to the pressure inside the chamber before opening the back pressure inlet valve.
13) Note that as the density of CO_2 can change drastically during the measurements, you might end up in the situation where CO_2 becomes heavier than your drop phase. If that happens the chamber needs to be turned upside down. This should only be done when the chamber is depressurized and cooled down.

End of measurements

1) When you are ready with your measurements, release the pressure inside the chamber by opening the back pressure outlet valve. Note that if very high temperatures have been used, you should let the chamber to cool down before releasing the pressure. Release the pressure from the front as well by opening the front pressure outlet valve.
2) Before disassembling the chamber, it is advised to do some initial cleaning of the 6-port valve, especially if samples like crude oil, have been used.

a. Rinse the 6 -port valve in both positions (1 & 2) by using carrier fluid pump. If needed, you can even increase the pressure in the pump before opening the dispenser inlet valve to push all the excess sample out of the tubes.
b. Rinse the valve also with appropriate solvent by pushing it through the sample introduction port with a syringe.

3) Disassemble the chamber by disconnecting all the tubes. Be careful in case of any liquid coming out while doing that.
4) When the system is shut down, you should first turn off the software, then Theta Flex and the electronic box.

4.4 Cleaning the system parts

Once the measurements are completed it is advised to clean the chamber and other parts of the system properly.

4.4.1 Cleaning the chamber

Disassemble the chamber and remove all the connectors attached by Teflon tape. Rinse the chamber with appropriate solvent depending on the used fluids. The chamber parts are stainless steel (EN 1.4401) and the windows are sapphire. Please refer to chemical compatibility chart if harsh chemicals need to be used for cleaning.

After initial rinsing, all the chamber parts and windows can be washed in dishwasher. The connectors and the needle should be washed by hand by pushing liquid through them. The bolts are not washed.

After washing in dishwasher, additional rinsing with suitable solvent and di-ionized water should be done.

4.4.2 Cleaning the six-port valve

It is advised to do an initial rinsing of the six-port valve as instructed in step-by-step instructions. This is especially important when highly viscous liquids like crude oil has been used in the valve. The six-port valve is cleaned by pushing through the appropriate solvent. The solvent used depends on the sample. The sample introduction port is PEEK, please refer to chemical compatibility chart if harsh chemicals has to be used. If necessary, the six-port valve can be detached from the instrument to do further cleaning under the fume hood for example. Also, the sample introduction port can be detached if necessary.

4.4.3 Cleaning the tubes on the valve panel

If only pure fluids are used in the pumps, it should not be necessary to clean the tubes in the valve panel. However, if contamination of the tubes is suspected, they should be cleaned by pushing appropriate solvent through the tubes. This can be done either with the syringe or with the help of manual pump.

4.4.4 Cleaning the manual pump

If only pure liquids are used in the manual pump, it should not be necessary to clean it. Also since the pump is mostly used as a carrier fluid pump or to pressurize the back of the chamber, minor

contamination should not be an issue. However, if needed, the pump can be cleaned by filling it several times with appropriate solvent. Rinsing with di-ionized water is recommended.

4.4.5 Cleaning the automated pump

If only pure liquids are used in the manual pump, it should not be necessary to clean it. However, if necessary, please refer the user manual of the automated pump for proper cleaning instructions.

4.4.6 Cleaning the automatic HPLC pump

If only pure liquids are used in the manual pump, it should not be necessary to clean it. However, if necessary, please refer the user manual of the automatic HPLC pump for proper cleaning instructions.

5 Maintenance and service

Only specifically approved spare parts are compatible with the chamber. Please contact your local Biolin Scientific provider for spare parts.

	Check	Replace
O-rings	After every use	When leaking occurs or the rings look damaged and at least after every five measurements
Windows	After every use	If any damage is detected and at least every 12 months
Bolts	When assembling the chamber, check that the threat looks undamaged and the bolt is easily rotated in place	If damage is detected or screwing the bolt becomes difficult and at least every 12 months
Teflon tape in connectors	-	If leakage on those parts is detected and whenever the chamber is cleaned
Swagelok connectors	In every assembly	If the connection feels jammed and at least every 12 months
Tubes		
Valves	As a part of yearly maintenance	If required
Over pressure valve	As part of yearly maintenance	If required
Over temperature switch	As part of yearly maintenance	If required

5.1 Making new connections

There might be a need to make new connections whenever the cleaning is done or one of the connections starts to leak. There are many types of connections in the system and below all of them are explained.

5.1.1 Connections to the chamber

There are five connection port to the chamber; three on top and two on the back flange. The connections are sealed by using Teflon tape. Two rounds of Teflon tape is rotated around the connectors in the direction of threads as shown in the Fig. 29.

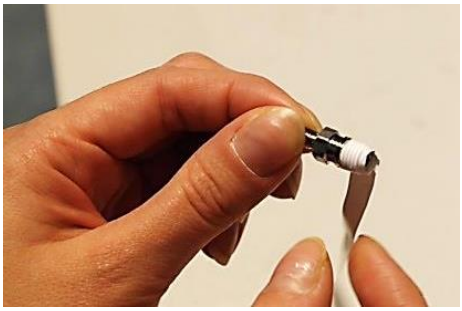


Fig. 29 Making the Teflon seal

The connector is then tightly attached to its place by using a spanner (Fig. 30). The caps can be attached the same way.

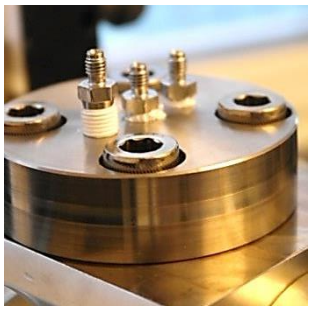


Fig. 30 Attaching the connector

5.1.2 Replacing tubes

Sometimes it might be necessary to replace one of the tubes in the system. The tube is cut with cutter tool (Fig. 31). Fasten tube at correct length by screwing the knob. Then start rotating the tool around the tube. Rotate a couple of rounds and then tighten the knob, and continue rotating. Repeat this until the tube is cut.

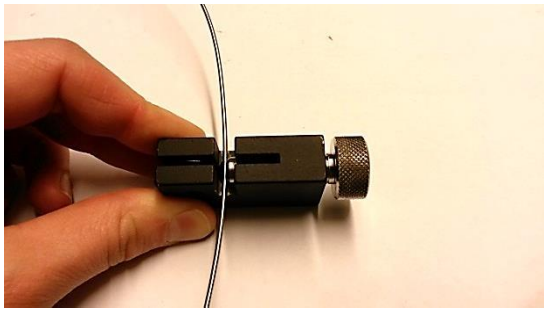


Fig. 31 Using tube cutter tool

The tube is connected using tube fittings and tee connectors. They contain two ferrules that form the connection to the tube when tightened (Fig. 32). Insert pre-cut tube through the cap nut into the tube fitting until it bottoms. Tighten the cap nut ¾ turns clockwise to create a tight connection. In Fig. 32 connection a tube to a needle valve is presented but same principle is used when making connection in the chamber.

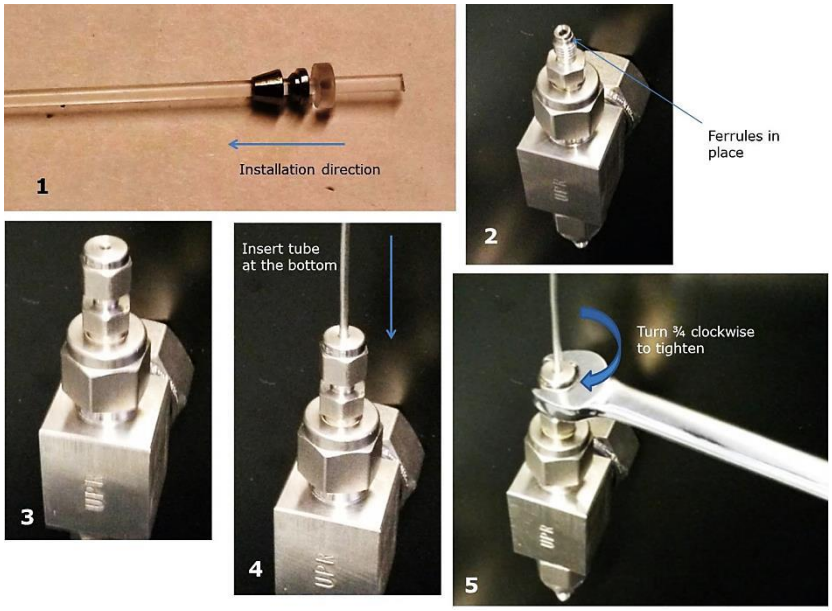


Fig. 32 Connecting tube to a tube fitting: Ferrules (1), Ferrules inside tube fitting (2), tube fitting in valve opening (3), Tube inserted (4), tightening the cap nut (5).

When the connection is opened again, the cap part is fixed on the tube and the NPT male connector is attached to a chamber / valve as shown in Fig. 33.

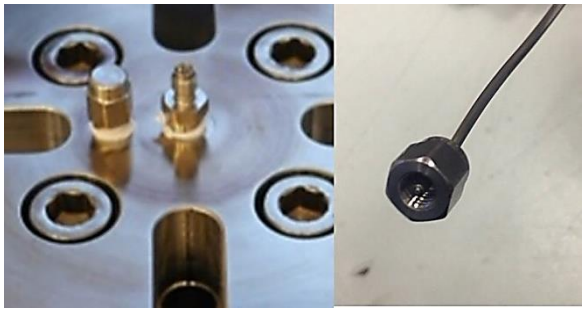


Fig. 33 NPT male connector on the chamber and the cap at the end of the tube

5.2 Mounting the needle valves

The back of the valve panel is shown in the Fig. 34.

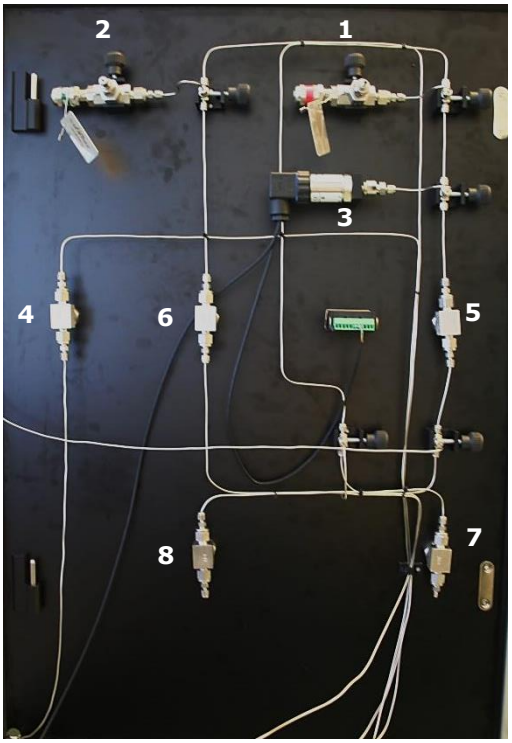


Fig. 34 Back of the valve panel. The panel contains the over pressure valves for front (1) and back (2) of the chamber, pressure transducer (3), Sample inlet valve (4), Front side inlet valve (5), Back side inlet valve (8), Front side release valve (7) and Back side release valve (8).

The needle valve parts are presented in Fig. 35. Insert valve body through the panel cut out from the inside and adjust to vertical position. For needle valves 4-6 in Fig. 34, the indicator arrow on the valve body must face **upwards**.

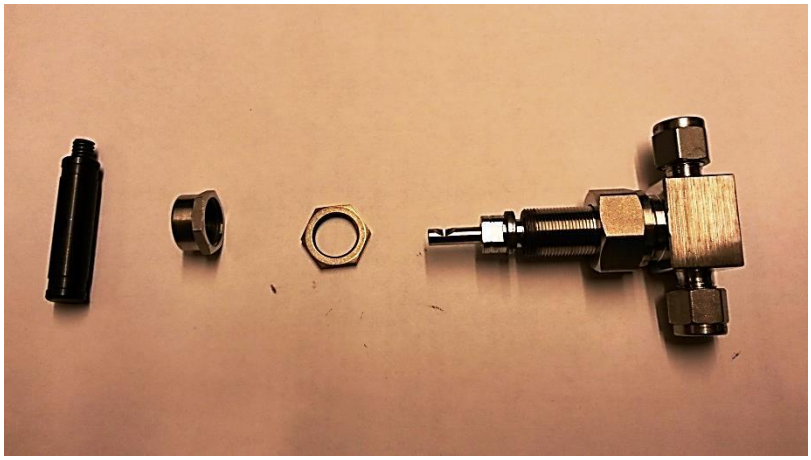


Fig. 35 Needle valve parts from left: handle, adjustment nut, fastening nut, valve body

For needle valves 7 and 8, the indicator arrow must face **downwards**. Add adjustment nut and tighten with wrench. Then insert handle and fasten with 1/8 inch hex key from the end (Fig. 36). Check the adjustment of the valve by rotating the handle; the valve adjustment should not be too tight or too loose. If too tight or loose, remove the handle and tighten or loosen the adjustment nut and repeat. When adjustment is finalized close the valve by turning all the way clockwise.

Fig. 36 Steps for mounting a needle vale; fastening nut, adjustment nut and handle

5.3 Changing the bolts

The bolts should be changed whenever damaged, when moving with high friction or at least every 12 months. The bolts are certified and meant to be used with pressurized vessels. When changing the bolts, they need to be greased for optimal functionality.

Use the grease included in the High Pressure Chamber delivery. Apply a thin layer of grease on each bolt and use paper to wipe out any excess grease. Use rubber gloves during the greasing to avoid skin contact.

5.4 Tool maintenance

The tightness of the chamber is assured by proper fixing of the bolts. The bolts are fixed using torque keys which ensure the correct amount of torque applied to the bolts.

The torque keys may wear over time giving incorrect torque. Therefore Biolin Scientific recommends replacing the keys with new ones once a year.

5.5 Pressure calibration

Pressure sensor is factory calibrated but in case it needs to be re-calibrated that can be done through OneAttension software. For proper calibration, an accurate external pressure meter is required. Pressure sensor of the automated pump can be utilized.

Open main menu at top left of the software and select device calibration. From the opening System Diagnostics window highlight the row with the name “Adda0” as shown in Fig. 37. Press calibrate.

A new pop-up window for pressure calibration will open as shown in Fig. 38. Pop-up window includes the instructions for the pressure calibration.

It is advisable to use relative large pressure range for the pressure calibration. The first pressure point can be atmospheric pressure but it is recommended that you use a slight overpressure as a first point, for example 5 bar. Increase the pressure to 5 bar with the pump, check the pressure reading from the external pressure meter. Write the pressure value to a upper left field named “point 1 pressure (bar)”. Click the upper “get current reading” button. Pressurize the system to a second pressure reading for example to 300 bar. Write the pressure value to lower left field named

"point 2 pressure (bar)". Click the lower "get current reading" button. When all four fields are filled, click calibrate and wait for the calibration to be done.

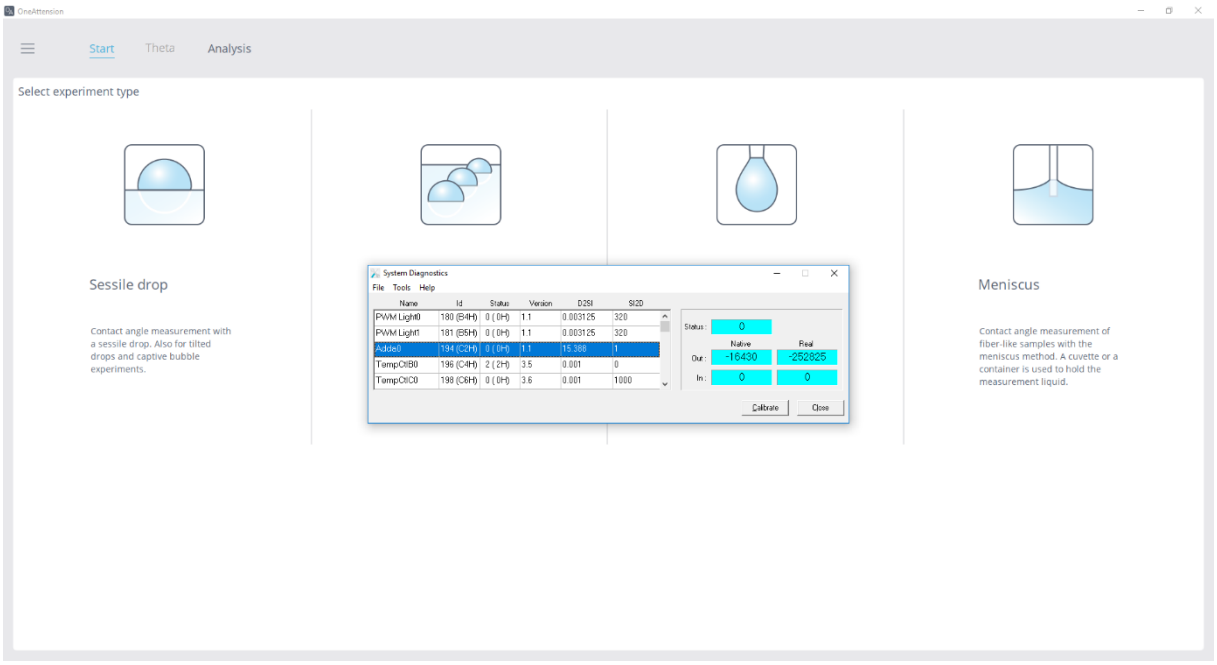


Fig. 37 Calibration of the pressure sensor is done from main menu -> Device calibration.

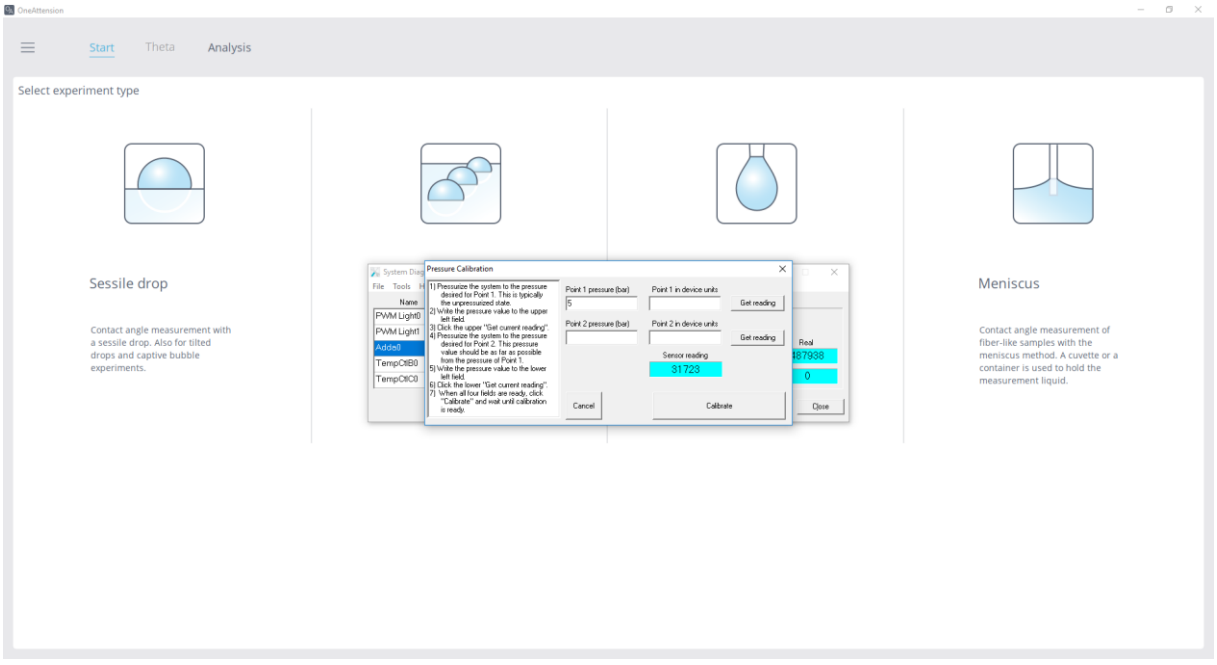


Fig. 38 Pressure calibration window. OneAttension guides through the pressure calibration.

6 Technical data

Specifications	
Measurement types	Interfacial tension, surface tension, contact angle
Possible phases	Gas – liquid, liquid-liquid, liquid-solid
Maximum pressure	400 bar
Temperature range	1 – 200 °C
Chamber volume	85 ml (without piston), 40-62 ml (with piston)
Pressure control	2 options: Pressure increase by piston or by adding more liquid/gas with pump
Dimensions	77 x 25 x 44 cm (L x W x H, Theta Flex + Chamber)
Weight	35 kg (Theta Flex + Chamber)
Voltage and current	100 – 240 VAC, 50 – 60 Hz, 1.8 – 4.2 A
Fuses	Mains: T5.0A 5x20 mm Heater: T25A 10.3x38.1 mm Peltier: T6.3A 5x20 mm
Environmental conditions	Ambient temperature: 15 – 30 °C Ambient pressure: 700 – 1060 hPa Ambient humidity: 20 – 80 % (non-condensing)

7 Contact information

If any problems arise please feel free to contact your local distributor or Biolin Scientific directly.

https://www.biolinscientific.com/contact-us

info@biolinscientific.com