

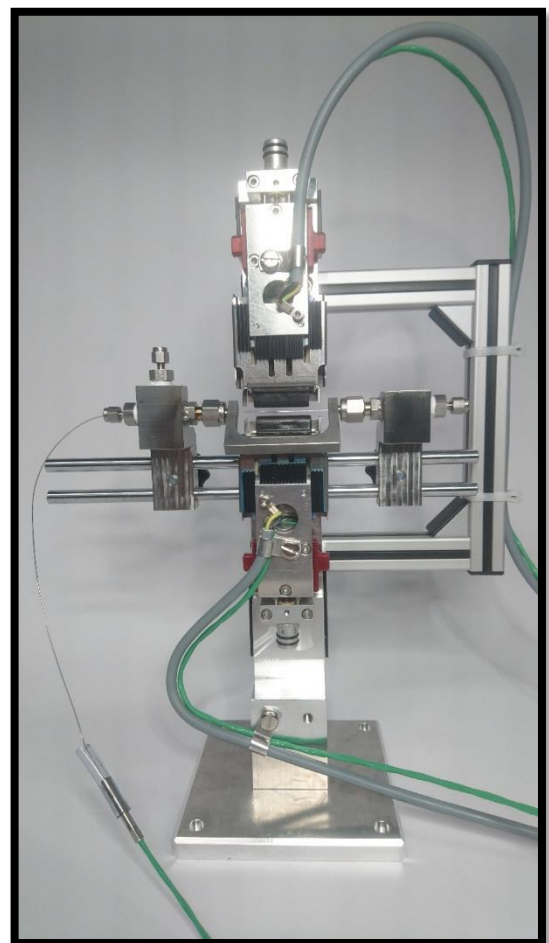
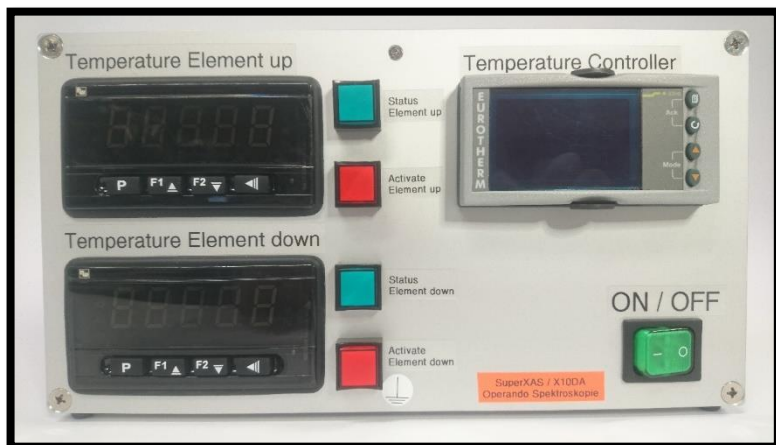
**Project:** SuperXAS

**Object:** Capillary reactor setup

**Document:**

# Operating manual for Capillary reactor setup

<b>Changes:</b>	1.0	30.09.2021	1st edition	Stephan Hitz Adam Clark, Olga Safonova, Stephan Hitz
	2.0	04.10.2021	Added feedback	



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# 1 Overview

The capillary reactor setup consists of a temperature controller and a holder for the capillary including two heating elements.

Two identical setups are available in the Operando Spectroscopy group.

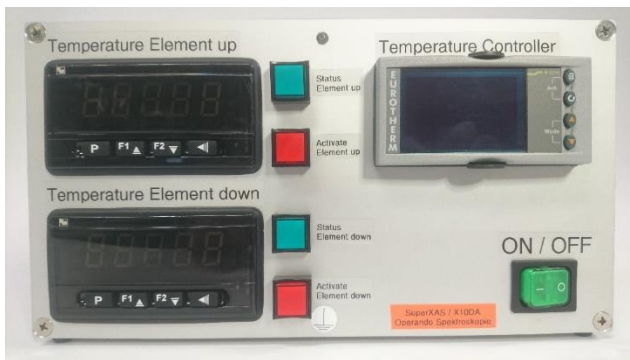


Figure 1: Front side of temperature controller

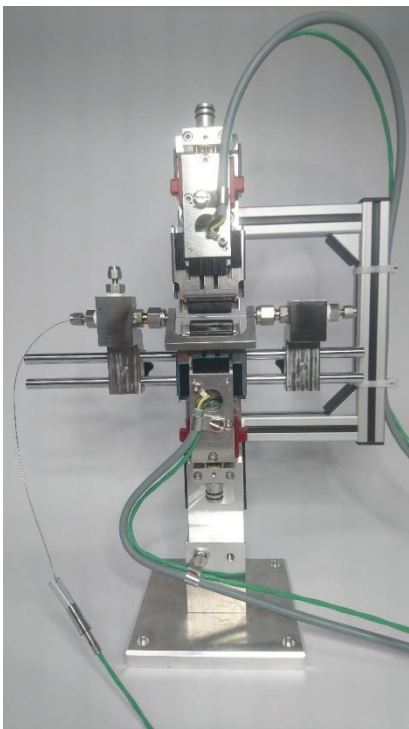


Figure 2: Capillary holder incl. heating elements

The size of the temperature controller box is 290 mm x 265 mm x 155 mm (L x W x H).

The size of the capillary holder is approx. 270 mm x 110 mm x 400 mm (L x W x H).

## 1.1 Connection diagram

The capillary holder can be separated from the temperature controller for transportation. To connect the capillary holder to the temperature controller, insert the corresponding plug into the socket of the temperature controller (each cable and socket is labeled).

The temperature controller can be connected to a computer and then be controlled with a dedicated software. Integration to the EPICS control system is possible.

On the experiment side, the capillary holder can be connected to gas lines, if required.

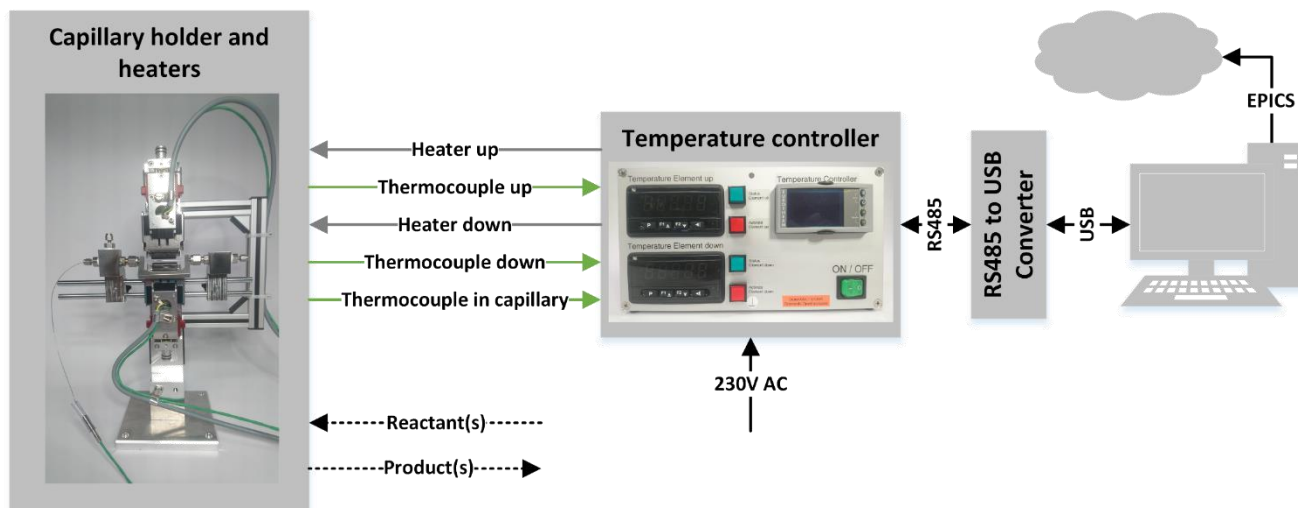


Figure 3: Connection diagram of the capillary reactor setup

## 2 Capillary holder

The capillary holder is used to mount and heat capillaries from  $\varnothing 1$  mm to  $\varnothing 3$  mm. The base plate can directly be mounted onto the sample manipulator of the SuperXAS/X10DA beamline.

### 2.1 Heating elements

Two heating elements, type T-MSH/30, from the company Elstein are used. The difference to the standard type T-MSH/20 is the increased length of the heater from 20 mm to 30 mm.

Property	Value
Type	T-MSH/30
Manufacturer	Elstein
Technology	Infrared heating element
Thermocouple	Integrated, type K
Power	80 W
Maximum temperature	860 °C (not recommended, see below)
Voltage	24 V

Table 1: Properties of a single heating element

For the capillary reactor setup, it is recommended to reduce the maximum temperature to 750°C, otherwise the lifetime of the heating elements will be significantly reduced.

The homogeneous heating zone is approx. 15 mm, as shown in Figure 4.

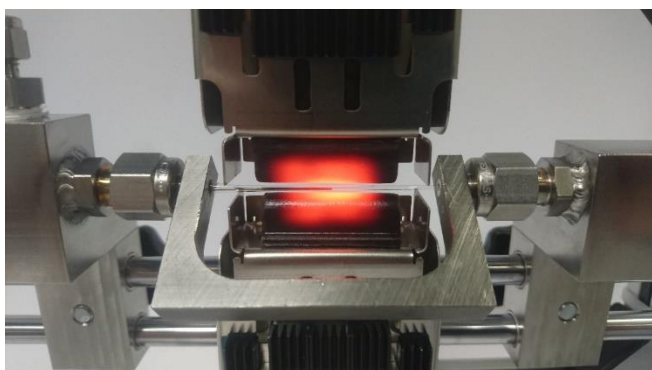


Figure 4: Heating elements in use at 750°C

## 2.2 Capillary mounts

Three different methods are available to mount capillaries to the holder.

To change the different mounts, unscrew the mount, lift it up and remove it from the holder. Add the new mount and screw it softly.

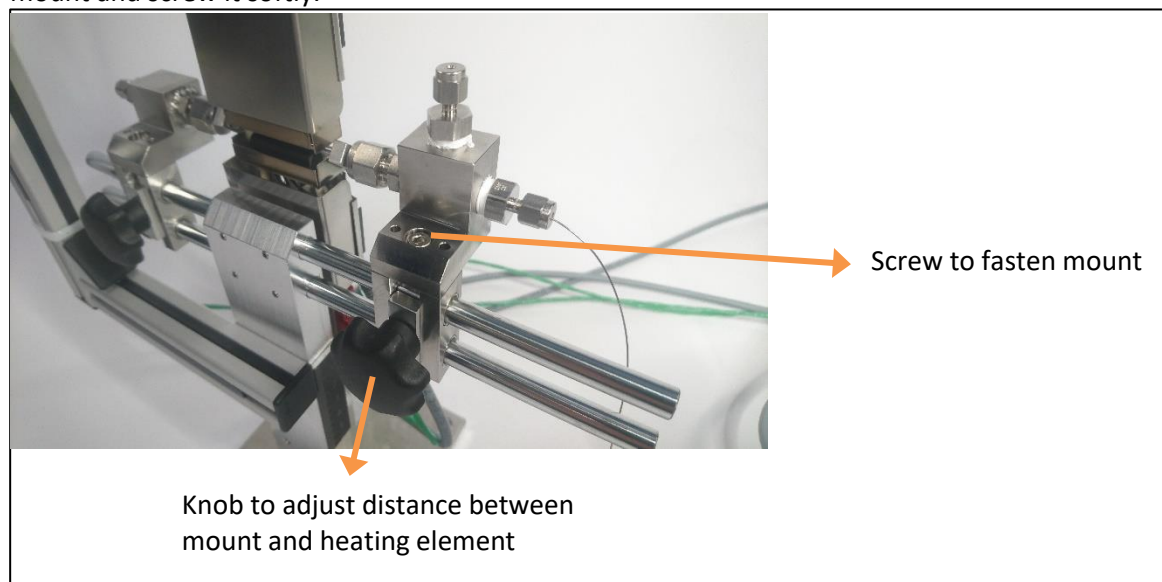


Figure 5: Mount adjustment and fixation

### 2.2.1 U-frame, standard method

A capillary is glued into a steel frame with "Araldite rapid". Gas connections are realized by 1/16" swagelok connectors. The thermocouple is inserted through a 1/16" Swagelok connector and sealed with a graphite ferrule.

The 1/16" connectors can be replaced in case of damage, order number SS-100-1-2RT from Swagelok.

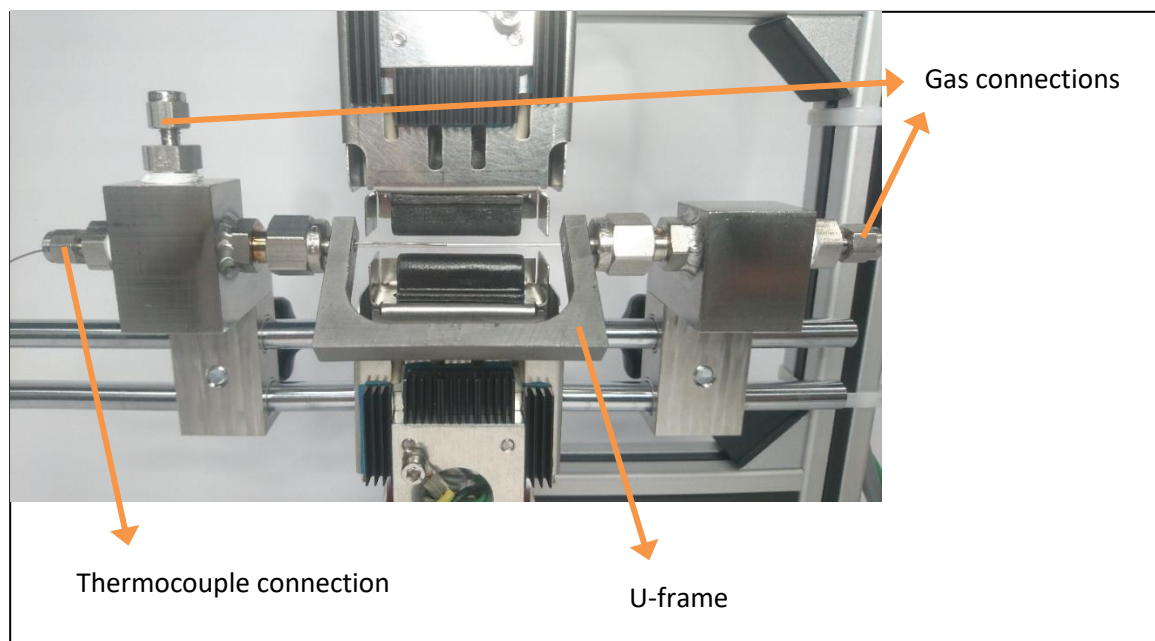


Figure 6: U-frame mount

## 2.2.2 Sealed capillary mount

Sealed capillaries (e.g. prepared under air-free atmosphere) are glued into a steel pipe. As no thermocouple exists in the sealed capillary, it is inserted only halfway into the heating zone, while an empty capillary is inserted from the other side. The thermocouple is inserted into the empty capillary.

The following preconditions should be met:

- Sample is at sealed end of the capillary (in the heating zone)
- Capillary sizes are identical
- If possible, the empty capillary should be filled with quartz-wool to simulate a sample

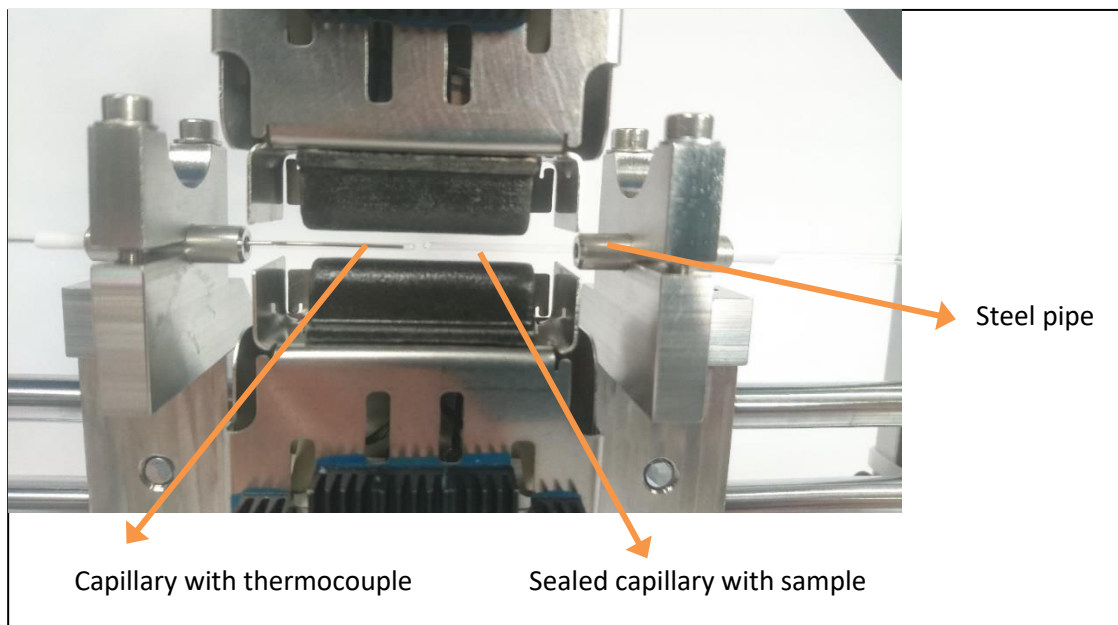


Figure 7: Sealed capillary mount

Steel pipes with an inner diameter of 2 mm, 3 mm and 4 mm are available.



Figure 8: Types of steel pipes incl. mount



### 2.2.3 Swagelok direct mount

If for example the sample inside the capillary must be stored under special atmospheric conditions (e.g. filled in the capillary under air-free atmosphere), additional valves are always connected to the U-frame. For this, the Swagelok nuts of the U-frame can be clamped.

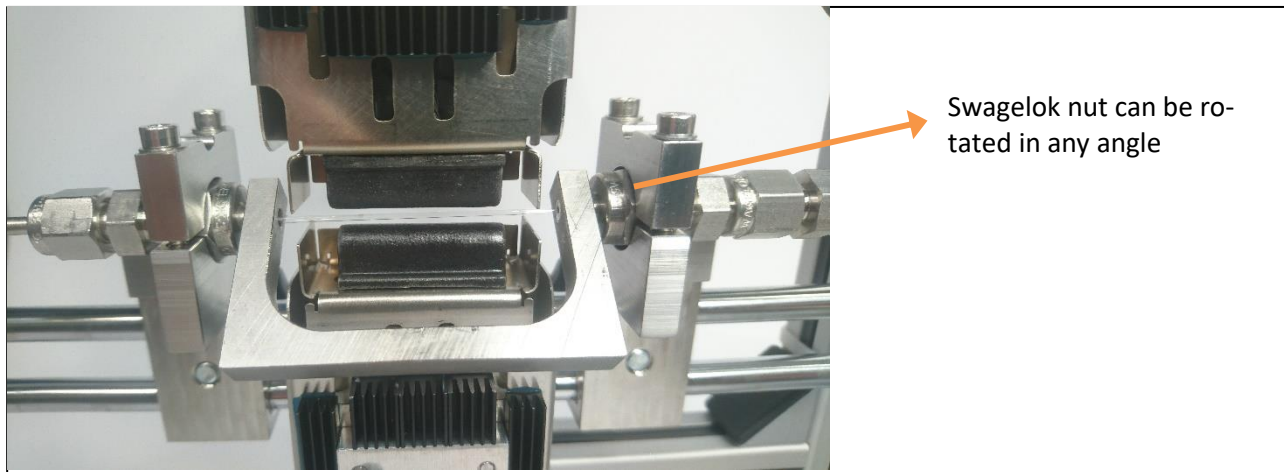


Figure 9: Swagelok direct mount

The U-frame consists of a 3/16" pipe including the ferrules and a 3/16" Swagelok nut.



### 3 Temperature controller

The temperature controller is a custom-made solution for the capillary reactor setup. It integrates the Eurotherm 32h8 controller, power supplies and heating element protection.

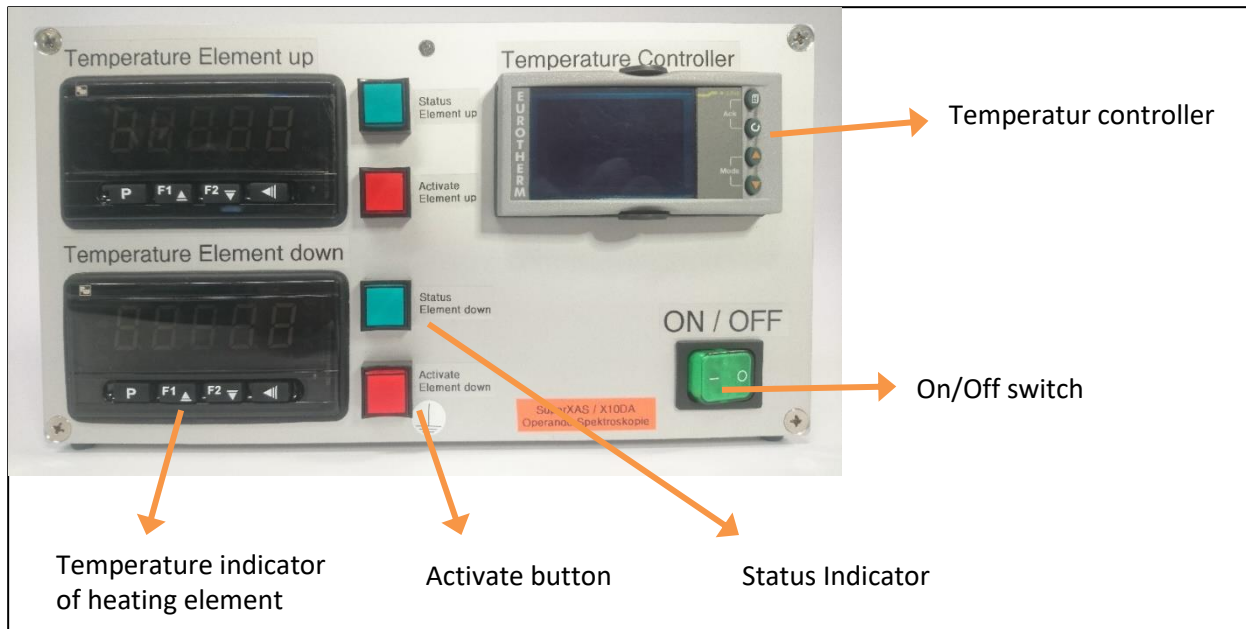


Figure 10: Front view of temperature controller

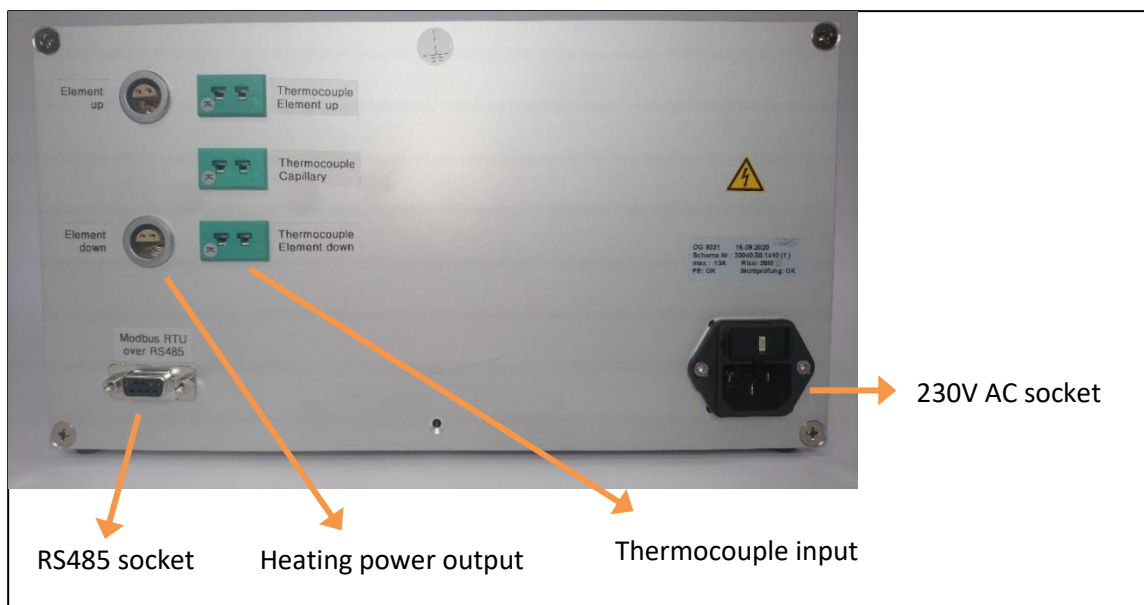


Figure 11: Rear view of temperature controller

### 3.1 Startup

- Connect all the cables. Attach capillary with samples. Place thermocouple next to the sample. Place both heating elements at about 0.5-1 mm next to the capillary
- Connect temperature controller to the mains
- Switch on the controller with the On/Off switch on the front
- Make sure that the setpoint of the temperature is below 20 °C and the actual temperature is close to room temperature
- Activate both heating elements with the push button, the status light must turn green

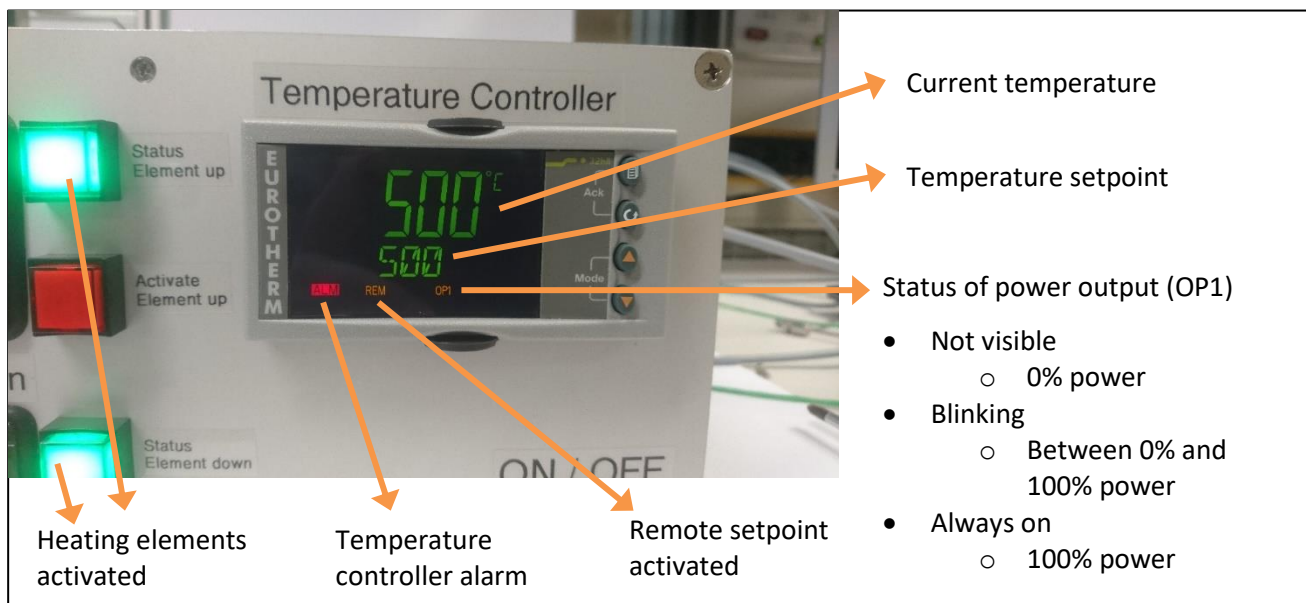


Figure 12: Display symbols of temperature controller

If the remote setpoint is activated, but no setpoint was (yet) set through the software, an Alarm is shown. This Alarm can be ignored; the controller is still working and can be used without the computer/software.

### 3.2 Heating element protection

Each heating element is protected against overtemperature. This can happen if the thermocouple inside the capillary is too far away from the heating element (>> 2 cm). The overtemperature limit is set to 750°C.

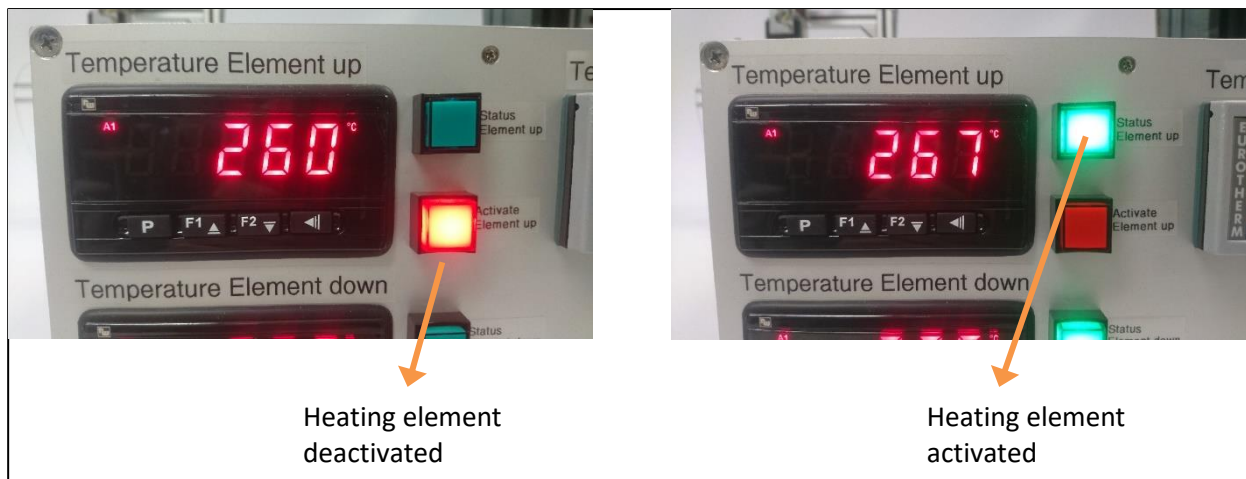


Figure 13: Heating element protection

### 3.3 Temperature feedback

To achieve the most accurate temperature inside the sample, please keep in mind:

- The thermocouple should be as near as possible to the sample
- The thermocouple should be inside the capillary. Placing the thermocouple outside, near the capillary, will not result in an accurate temperature measurement!
- Use a thin thermocouple to reduce its thermal mass, preferably  $\varnothing 0.5$  mm or thinner
- Always use a type K thermocouple!
- It is possible to use a thermocouple of one of the heating elements to control the temperature. Due to the technology of the heaters (IR-radiators), the airflow around the heaters and other factors, this can result in temperature deviations between the heater and the sample of  $\gg 100^\circ\text{C}$ .
- For best accuracy, calibrate the PID settings of the controller. See chapter Software.

## 4 Software

A dedicated python script with graphical GUI is available for the capillary reactor setup. It has the following functionalities:

- View temperature curve (setpoint and actual) and output power in realtime
- Change setpoint temperature, ramp rate and PID settings
- Log temperature curve and save to file
- Run simple recipes with temperature profiles
- Auto-Calibrate controller
- Host epics server for integration in epics environment

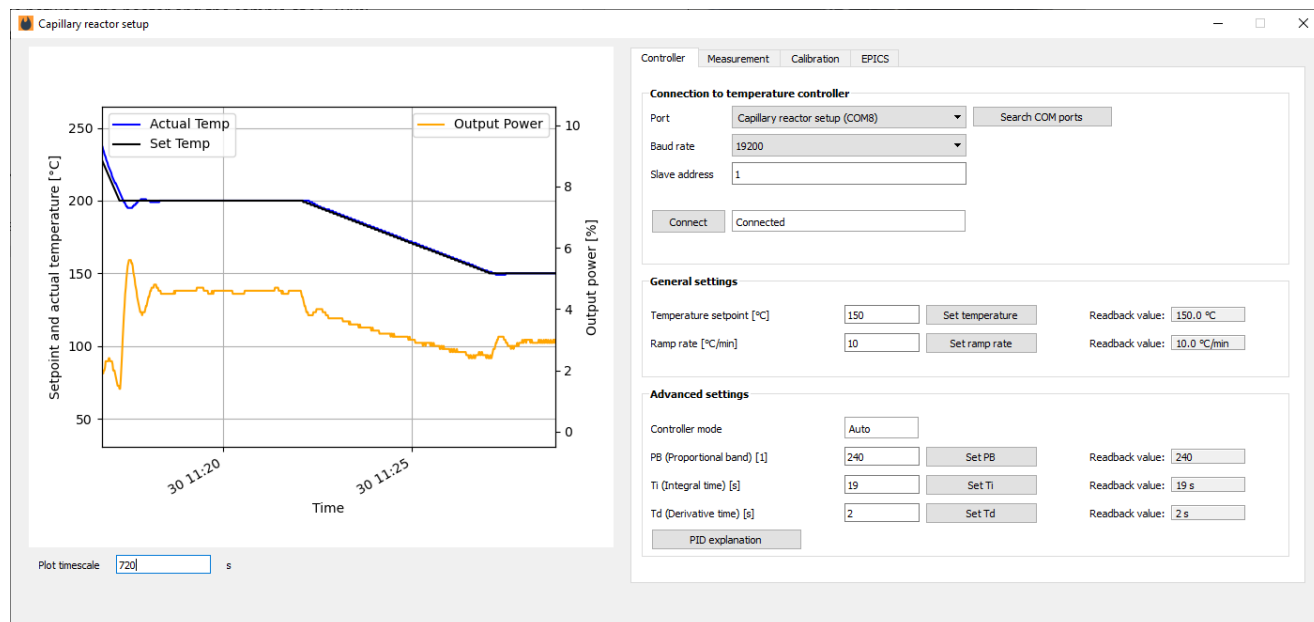


Figure 14: Graphic user interface

Where necessary, functionalities are described how to use them.

### 4.1.1 Setting PID values

If needed, the PID settings of the controller can be adjusted. They can either be adjusted manually or through a calibration process. During the calibration, the software will heat the capillary in two steps and calculate the PID values according to the measurements. This calibration process is the recommended way to change the PID values, as no knowledge about control systems is needed. However, fine PID adjustments may be possible to achieve best results. The basic principle is (text also available in the software):

- The PB value (Proportional band) is the most important value. It defines how aggressive the controller reacts. The bigger the PB value, the LESS aggressive the controller will react.
- The Ti value (Integral time) is used to reach the desired setpoint. The difference between the set temperature and the actual temperature is integrated over time and applied to the output of the controller. Increasing this value will SLOW DOWN the controller, it takes longer to reach the set temperature.
- The Td value (derivative time) is used to react fast to disturbances. The derivative of the actual temperature is applied to the output of the controller. Increasing this value will INCREASE the response on disturbances.
- If you detect oscillations in your actual temperature, first set Td to zero and then start to increase PB. If necessary, also increase Ti. If everything looks stable (no oscillations), start to slowly increase Td.

### 4.1.2 EPICS interface

An epics interface is available with basic functionalities. The following channels are available:

- X10DA-EH-CRS:TEMPCONTROLLER.SETTEMP (read/write)
- X10DA-EH-CRS:TEMPCONTROLLER.RMP-RATE (read/write)
- X10DA-EH-CRS:TEMPCONTROLLER.MODE (read)
- X10DA-EH-CRS:TEMPCONTROLLER.ACTTEMP (read)
- X10DA-EH-CRS:TEMPCONTROLLER.CTRLTEMP (read)
- X10DA-EH-CRS:TEMPCONTROLLER.OUTPOW (read)

The beamline prefix can be changed between “X10DA” and “X01DA”, depending the beamline where the setup is used. If the values of the two writable epics channels are changed (through epics), it will be recorded in the status field.

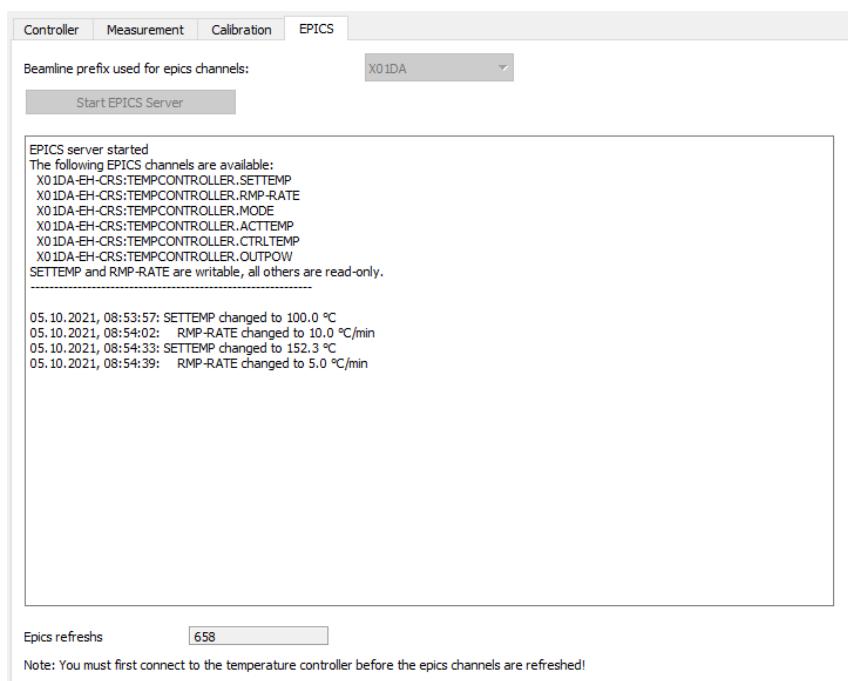


Figure 15: Epics subpanel

## 4.2 Connection to temperature controller

A “RS485 to USB Converter” is necessary to connect the temperature controller to a computer. The setup is tested with the EX-1303 converter from EXSYS, but should work with any “RS485 to USB Converter” if the pin-assignment on the 9-pin Sub-D is correct.

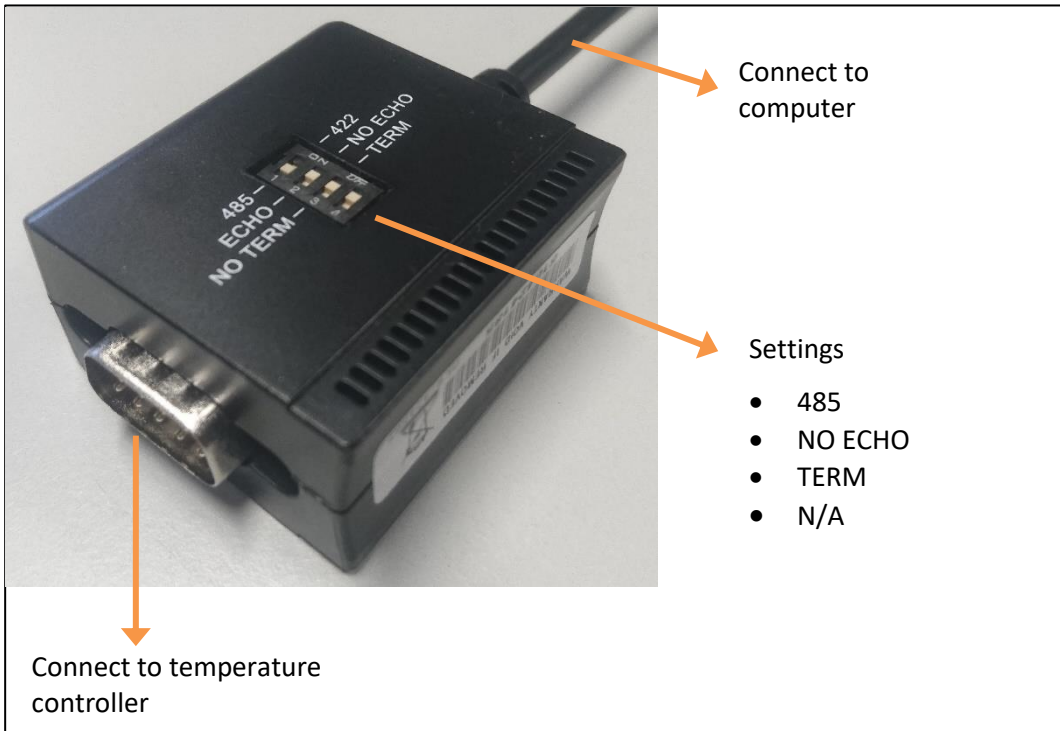




Figure 16: USB to RS485 Converter, EX-1303

A driver needs to be installed on windows computers, please visit the EXSYS Website for the newest driver or search on Google for the driver installation file “CDM21228\_Setup.exe”.

## 5 Safety

The following safety aspects must be taken into account.

Hazard	Explanation
	Do not open the temperature controller box! Call a technician if the device is not working properly.
	Do not touch parts of the heating elements and it's surrounding. Cool down the setup before moving it around.