



SAE N₂O Isotope Lab

Documentation for TREX-QCLAS



Kristýna Kantnerová
kristyna.kantnerova@usys.ethz.ch
kantnerova.kristyna@gmail.com

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1 Introduction

This is a documentation of the ETH-TREX (TRace gas EXtractor) that was built between 2019 and 2020 by Erkan Ibraim and finished and coupled with the QCLAS (quantum cascade laser absorption spectroscopy) instrument by Kristýna Kantnerová. The purpose of the document is introducing the TREX-QCLAS to the SAE team members and to provide a manual for anyone who is supposed to work with it. Figure 1.1 shows the system when it was employed in the B32 lab of the LFW building.

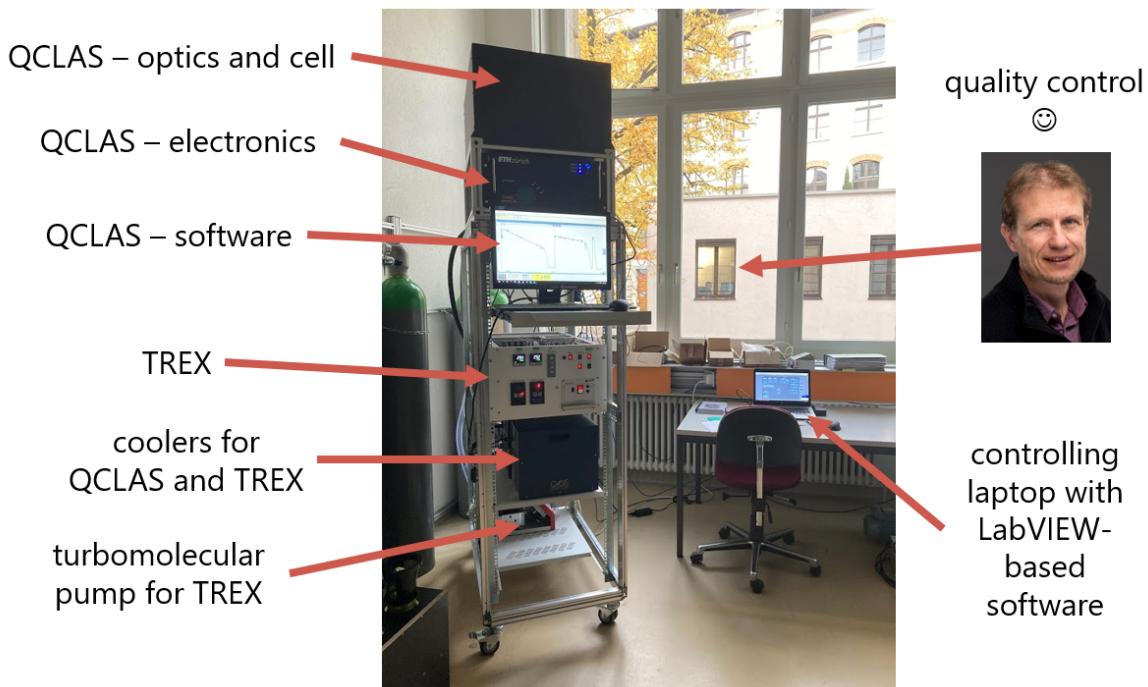


Figure 1.1: Parts of the TREX-QCLAS system.

First, the preconcentration unit TREX and its operating software TrexOS are described. In the second part, there is information about the QCLAS instrument and the related TDLWintel software. The third part presents the whole TREX-QCLAS system. Then, the preconcentration routine is described in a detailed way. Finally, issues that can commonly occur are summarized together with troubleshooting tips, and devices that should be regularly maintained are specified.

This documentation is available as an [Overleaf project online](#) or as a pdf file on the P: drive of the SAE group (P:/users/Kristyna/ETH-TREX).

1.1 Brief information on the system

The TREX-QCLAS system is used for high-precision measurements of preconcentrated N₂O samples in the batch mode. The system is operated through a LabVIEW-based software called "TrexOS" on a lab notebook and the TDLWintel software on the QCLAS computer. The TREX unit performs cryogenic adsorption of N₂O from sampled gas/air into a stainless-steel trap, separation of co-adsorbed species during step-wise heating and purging by synthetic air (SA), and finally desorption of N₂O into an optical cell of the QCLAS instrument. It is crucial to match exactly the peak of N₂O desorption in order to provide full recovery of adsorbed N₂O and the volume of SA needed to desorb the N₂O can be kept as low as possible. The volume of ambient air that is needed in order to obtain sufficient N₂O concentration in the cell has to be determined experimentally, as the exact volume of the QCLAS cell is not known. For the QCLAS analysis, the preconcentrated N₂O amount needs to correspond to the concentration of used reference gases.

More detailed description of the TREX-QCLAS system can be found in Chapter 4.

2 Description of TREX and TrexOS

This part describes the physical appearance of TREX, all individual components, and the operating TrexOS software written in LabVIEW.

2.1 TREX – TRace gas EXtractor

The abbreviation TREX stands for the TRace gas EXtractor. Trace gases are defined as gaseous components of the atmosphere other than nitrogen, oxygen, and argon. The TREX is based on liquid nitrogen-free thermal separation of target trace substances from sampled air. Mohn et al., 2010 reported on the application of this approach by utilizing a strong Stirling cryocooler.

The analysis of trace gases is limited by their low abundance in ambient air. However, by separating trace gases from large volume of ambient air and subsequent diluting with synthetic air, samples with enriched abundance of the analyzed trace gas with a defined matrix can be achieved, thereby allowing high-precision analysis. Following Mohn et al., 2010, Eyer et al., 2015 developed a more powerful and more compact preconcentration device. This allowed extracting CH₄ from ambient air. Subsequently, Ibraim et al., 2017 presented a similar device that was able to extract N₂O. Currently, ETH Zurich is the first institute outside of Empa running a TREX-QCLAS instrument. This instrument is based on a copy of the TREX device that was used in the work by Ibraim et al., 2017.

2.1.1 Diagrams and flow schemes

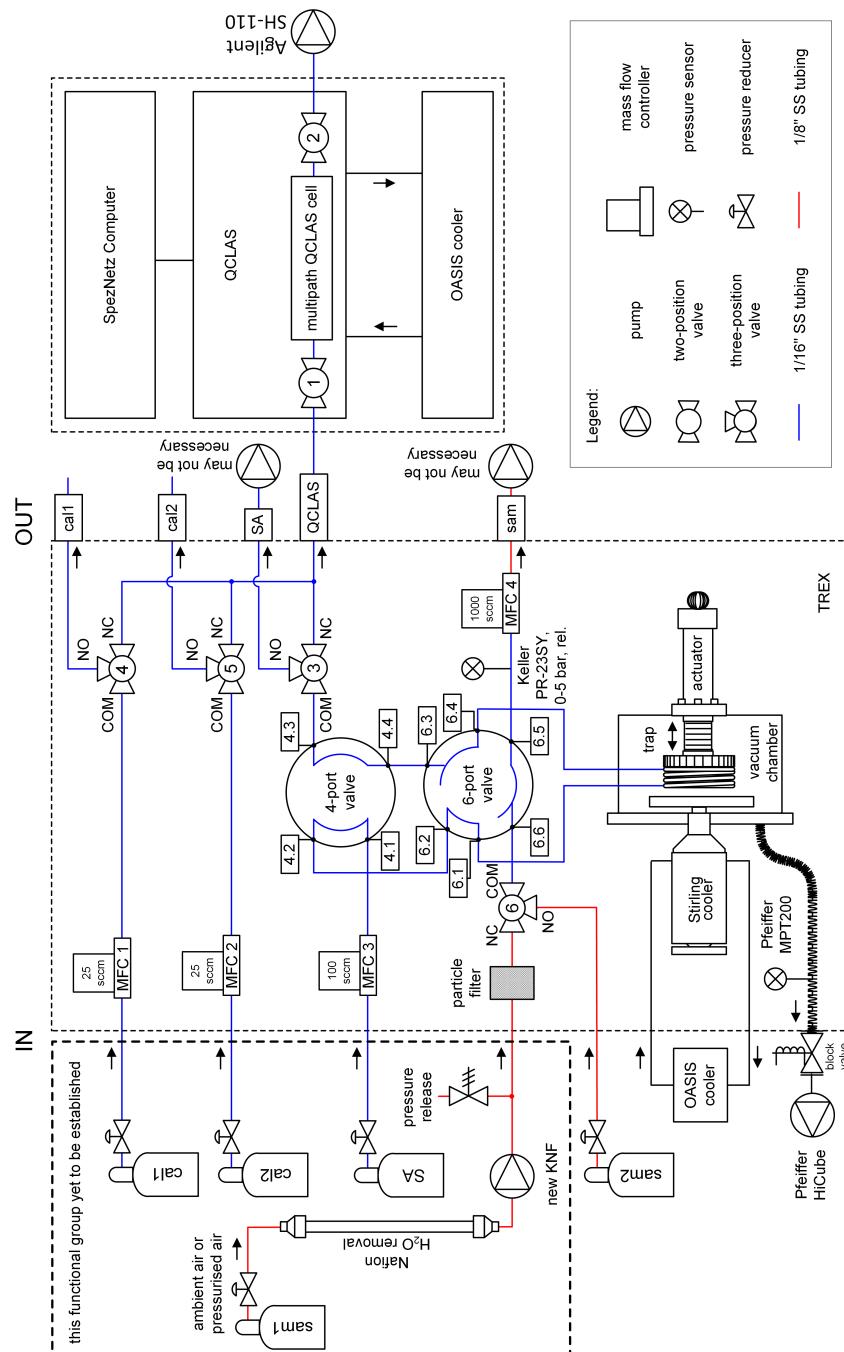


Figure 2.1: A flow scheme of the TREX unit.

2.1.2 Components

The information in this section was collected from the knowledge on this TREX unit and a technical documentation of another TREX unit at Empa Dübendorf (the two-trap TREX for clumped methane). Therefore, it may happen that some information is incorrect (especially electrical connections), even though I tried my best to check it with this design.

Mass flow controllers (MFCs)

The MFCs are daisy chained and connected via a single cable to the Moxa board. The original RS-485 DB9 was modified to a RJ45 connector according to Fig. 2.2 and Table 2.1. The pins 2 and 3 should be connected to the 24 V-power supply.

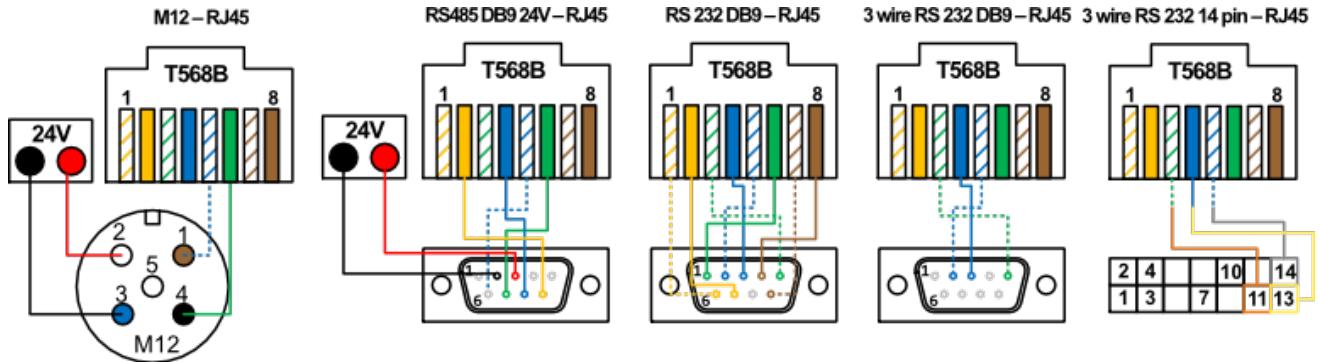


Figure 2.2: Pin mapping for RJ45 connectors.

Table 2.1: Pin mapping for RJ45 connectors.

Moxa	RJ45 pin	RS-485 DB9 24 V	RS-232 DB9	3-wire RS-232 DB9
DSR	1 →	–	6	–
RTS	2 →	9	7	–
GND	3 →	–	5	5
TxD	4 →	8	3	3
RxD	5 →	6	2	2
DCD	6 →	7	1	–
CTS	7 →	–	8	–
DTR	8 →	–	4	–
–	24 V	3	–	–
–	GND	2	–	–

VICI valves

There are two VICI valves – a 2-position 4-port valve and a multi-position 6-port valve. The number of positions for the 6-port valve can be set from 2 to 96 (see its manual). In TREX, it is set to a total of 12 positions. Figure 2.3 shows how the ports are connected in the individual positions. Some positions of the 6-port valve result in the same port connections, therefore there are only five possible states, as Table 2.2 shows.

The cable for serial connection of the VICI valves to the Moxa board is self-manufactured. The original 3-wire RS-232 DB9 was modified to a RJ45 connector according to Fig. 2.2 and Table 2.1. The communication with the VICI valves can be tested via the HyperTerminal software (`hypertrm.exe`) on the lab notebook. A communication file used for the initial testing can be found under `C:\Users\localadmin\Documents\Technics\ETH TREX\Vici.ht`. Commands to communicate with the valves via HyperTerminal can be found in the VICI manual.

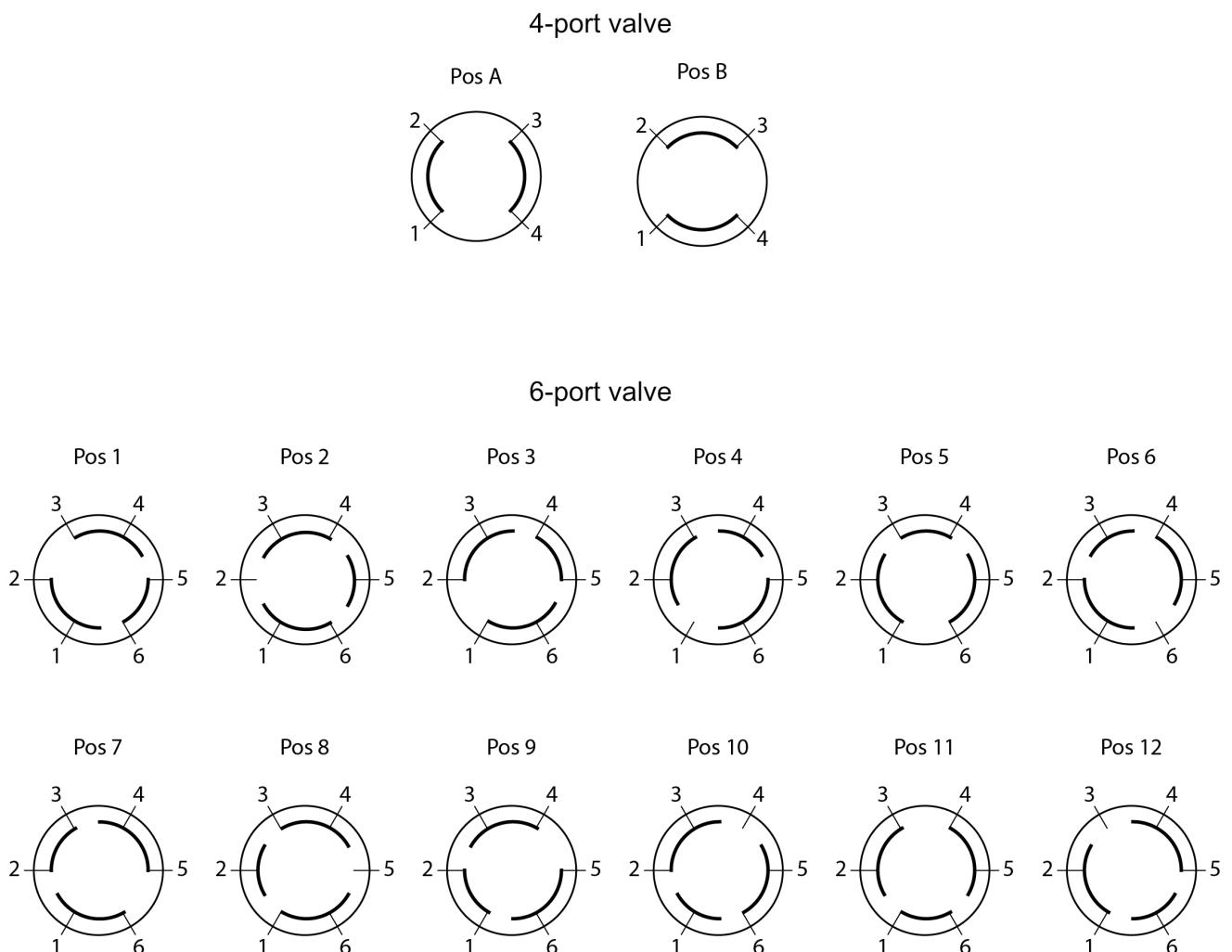


Figure 2.3: Diagrams showing the positions of the VICI valves and the resulting connected ports.

Table 2.2: Port connections for the 6-port VICI valve set to a total of 12 positions. 1 means an open port connection, 0 means a closed port connection.

port connection	position				
	1/5/9	2/8	3/7/11	4/10	6/12
1–2	1	0	0	0	1
2–3	0	0	1	1	0
3–4	1	1	0	0	0
4–5	0	0	1	0	1
5–6	1	0	0	1	0
6–1	0	1	1	0	0

Linear actuator

The linear actuator is connected to the SimStep control box. The cables for serial connection of the linear actuator to the Moxa board are self-manufactured. The original RS-232 DB9 was modified to a RJ45 connector according to Fig. 2.2 and Table 2.1.

The communication with the linear actuator can be tested via the HyperTerminal software (`hypertrm.exe`) on the lab notebook. A communication file used for the initial testing can be found under `C:\Users\localadmin\Documents\Technics\ETH TREX\SimStep.ht`. Commands to communicate with the linear actuator via HyperTerminal can be found in the device manual.

There are three positions of the actuator that are important: 0 – the trap has no contact to the base plate; 3500 – the first thermal contact between the trap stand-off and the base plate, temperature dropping by $0.1 - 0.2 \text{ }^{\circ}\text{C}/\text{s}$; 6400 – the trap is fully pressed against the base plate, the cooling of the trap has the highest rate. In Erkan's older routines, he was using position 6500 but he told me 6400 when we met. However, those extra 100 points should not make any difference so I stick to 6400 points.

In case the linear actuator needs to be realigned, follow these instructions:

1. Determining the position with the first thermal contact:
 - (a) Move the trap slowly to the base plate (with steps of 100 points each) and note down the position where the trap temperature starts dropping quickly.
 - (b) Subtract 3500 from this position and move the trap to that (the zero position).
 - (c) Switch off the SimStep control box on the TREX front panel.
 - (d) In TrexOS, stop everything and exit the software.
 - (e) Switch on the SimStep control box.
 - (f) Restart TrexOS and reconnect all components.
2. Alignment of the stop buttons:
 - (a) When the actuator is at the zero position, realign the right button such that the metal part of the button has a contact with the moving pin but the stop button is not activated (but further movements in the negative direction, away from the base plate, would activate the stop button).
 - (b) Move the actuator to the 6400 position.
 - (c) Realign the left button such that the metal part of the button has a contact with the moving pin but the stop button is not activated (but further movements in the positive direction, to the base plate, would activate the stop button).

JUMOs

There are two JUMO temperature controllers – JUMO 1 measures the trap temperature and actively heats the trap if needed using a PID controller, JUMO 2 only measures the temperature inside the TREX unit (but it is also a fully functioning controller, just not used). The PID settings were set according to the file in `C:\Users\localadmin\Documents\ETH TREX-QCLAS\Jumo_Einstellungen_CH4_TREX.xlsx`. On the TREX front panels, two displays show current values (the upper row) and set values (the lower row) that are accessible via TrexOS.

The cable for serial connection of the JUMOs to the Moxa board is self-manufactured. The original 3-wire RS-485 DB9 was modified to a RJ45 connector according to Fig. 2.2 and Table 2.1. The communication with the JUMOs can be tested via the HyperTerminal software (`hypertrm.exe`) on the lab notebook. A communication file used for the initial testing can be found under `C:\Users\localadmin\Documents\Technics\ETH TREX\Jumo.ht`. Commands to communicate with the valves via HyperTerminal can be found in the device manual.

Parker valves

There are six Parker valves – four 3-way valves and two 2-way valves. They are connected through two ICP modules (one module has five channels only) and the L611 valve driver (Fig. 2.4). The L611 was designed at Empa. It is connected according to Table 2.3. Pins 1 – 6 are connected to the negative connection on the ICPs. All positive pins on the ICPs are connected together in series and connected to pin 7.

Two external valves can be connected via the RS-232 connectors on the TREX back panel (dummies ready to be used, not connected inside TREX). Connect the valves according to Table 2.4 and Fig. 2.5.

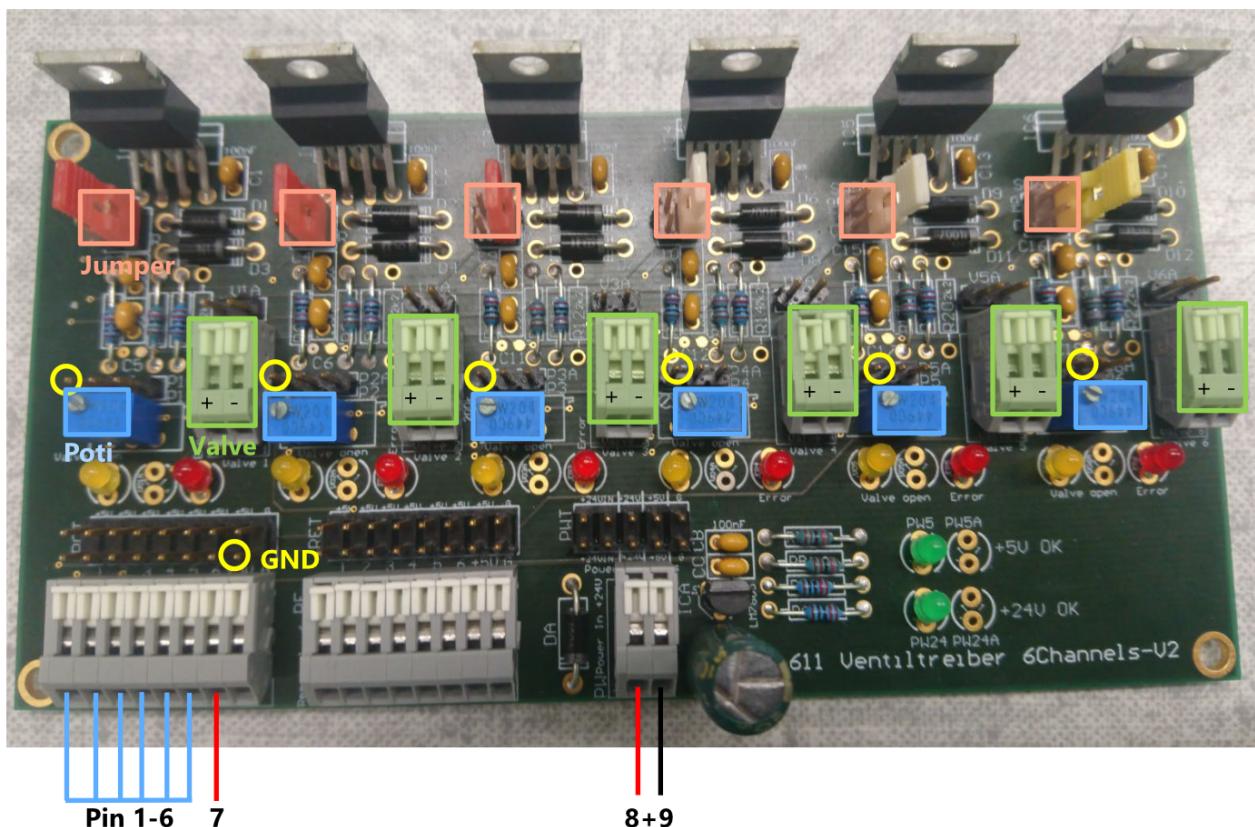


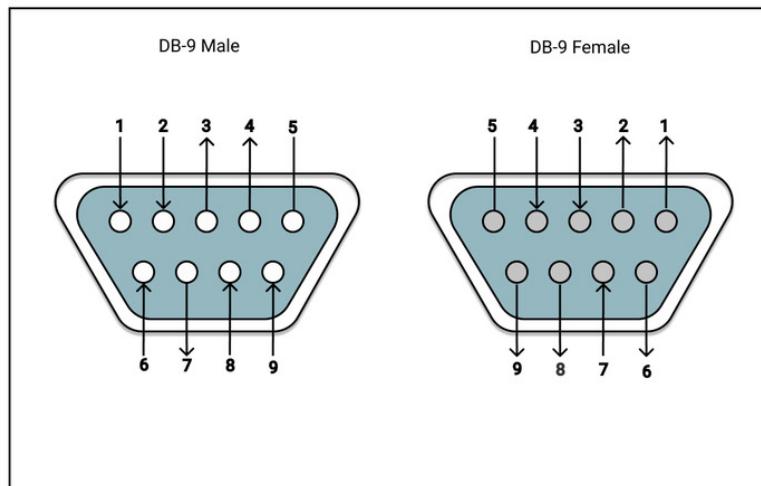
Figure 2.4: The L611 valve driver. More information in Table 2.3.

Table 2.3: The setup of the L611 valve driver

location on L611	function
pin 1 – 6	relais 1 – 6 (-)
pin 7	5 V
pin 8	24 V
pin 9	GND
valve 1 – 6 (green)	valves 1 – 6, no polarity
potentiometer 1 – 6 (blue)	adjust valve-hold voltage to 12 V
jumper 1 – 6 (red)	put jumper to connect the two left pins
GND 1 – 6 (yellow)	–

Table 2.4: Pin allocation for connection of external valves to the RS-232 DB9 connector

external valve	1		2	
	+	-	+	-
polarity	1	6	5	9
pin colour	orange	brown	purple	yellow

**Figure 2.5:** Pin allocation of a male and female RS-232 DB-9 connector, from virtual-serial-port.com (dummies ready to be used, not connected inside TREX).

Stirling cooler

The Stirling cooler is connected via a control unit to two 48 V DC power supply units (PSUs) with a provided cable. The temperature sensor of the cooler has 4 cables and is connected via a connector through the vacuum chamber to the TEMP connector of the control unit. The control unit communicates via a RS-232 14 pin cable that was modified to a RJ45 connector according to Fig. 2.2 and Table 2.1. The communication with the Stirling cooler can be tested via the HyperTerminal software (`hypertrm.exe`) on the lab notebook. A communication file used for the initial testing can be found under `C:\Users\localadmin\Documents\Technics\ETH TREX\Cryocooler.ht`. Commands to communicate via HyperTerminal can be found in the device manual.

A flow meter/shut-off can be connected between pins 7 and 10 of the control unit. The lateral load of the cold tip must not exceed 30 N, the axial load must not exceed 300 N. The engine of the Stirling cooler requires external cooling (one of the OASIS coolers), with a flow rate at least 15 mL/s and inlet temperature between 10 °C and 55 °C.

Pressure sensors

There are two pressure sensors – Keller 23-SY that measures relative pressure in the trap, and Pfeiffer MPT 200 that measured absolute pressure in the vacuum chamber.

Keller 23-SY is connected via a RS-485 cable that is connected to one of the ICP modules, and then via a RJ45 cable. The pressure range is between 0 and 5 bar.

Pfeiffer MPT 200 is connected via a M12 cable that was modified to a RJ45 connector according to Fig. 2.6, Table 2.5, Fig. 2.2 and Table 2.1. The pressure range is $5 \cdot 10^{-6}$ – 1 bar ($5 \cdot 10^{-9}$ – 1000 mbar).

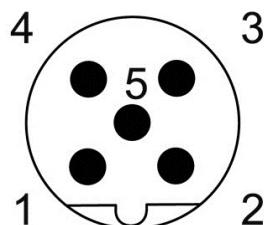


Figure 2.6: Pin allocation of a M12 plug.

Table 2.5: Pin allocation for Pfeiffer MPT 200

pin	function	wire colour
1	RS-485 D+	brown-blue/white
2	24 V	white
3	GND	blue
4	RS-485 D-	black-green
5	unused	–

Coolers

There are two external cooling units for the Stirling cooler and the QCLAS instrument. The units are set to 20 °C and 14 °C, respectively. The cooling liquid for the OASIS coolers consists of 80 vol. % H₂O and 20 vol. % ethanol.

Vacuum pumps

There are two vacuum pumps – Agilent SH-110 that is connected to the QCLAS outlet, and Pfeiffer HiCube 80 Eco that is connected to the vacuum chamber.

Moxa box

There is a serial port server Moxa NPort 5650-8-DT-J that serves for connecting all components to the lab notebook and the TrexOS software. It can process RS-232, RS-422, and RS-485 in different configurations. The ports on the Moxa box are of the RJ45 type, therefore the original cables of the components were modified. The components are connected according to Fig. 2.7.

The Moxa box is connected to the lab notebook via a RJ45 cable. It can be configured via its softwares NPort Administator and Nport Windows Driver Manager after unlocking. The login is "admin". The password was changed from "moxa" to "moxa.1234" as the Moxa software required a more secure password. After the password was changed, the Moxa box restarted itself but afterwards did not accept neither the old nor the new password. The password can be changed only by resetting the Moxa box back to factory settings. However, it is usually not needed to be able to access the Moxa settings as everything has already been set and works fine, so the password has not been reset.

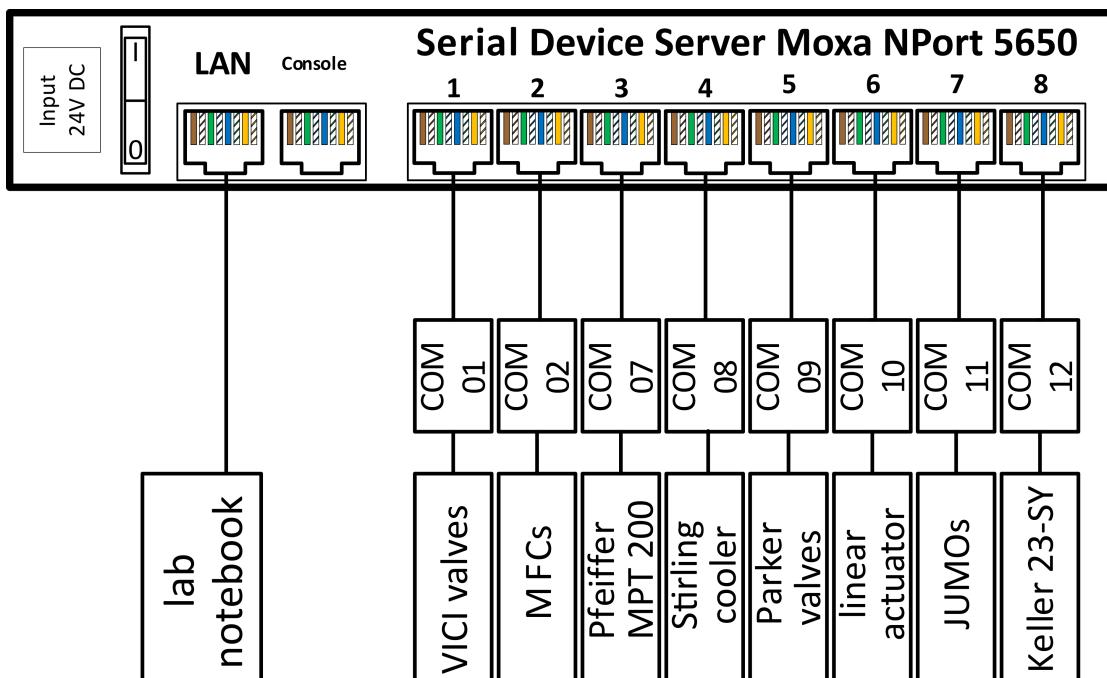


Figure 2.7: Connection of the components to the Moxa box.

Other electrical parts

Power supply units

The TREX is powered by a 230 V power supply with a fuse. Inside the TREX, there are two voltage circuits – 24 V and 48 V (Fig. 2.8 and Fig. 2.9).

Axial fans

There are two 24 V DC axial fans in order to remove heat produced by the components. The fan mounted on the back panel is used to suck in fresh air and the one that should be on the side will blow out air from the inside.

LEDs

Originally, there were two big LEDs that died on 06/10/2020 – they were on and the trap was moved a bit (by 500 points), which resulted in a shortcut that killed the LEDs. Andreas Schneider from LPC (D-CHAB) exchanged them on 14/12/2020 for a series of six blue LEDs.

Mechanical parts

Vacuum chamber – tube feed-through

There are two feed-throughs for gas tubes that go through the vacuum chamber – trap inlet and trap outlet. The 1/16 inch-gas tubes go through the wall of the vacuum chamber inside two Swagelok ISO connectors.

Vacuum chamber – electrical feed-through

There are 10 electrical connections that go through the vacuum chamber via a feed-through connector. Each pin is used according to Fig. 2.10.

Vacuum chamber – leak rate

The leak rate of the vacuum chamber was measured last time on 05/02/2021. It was determined to be 2.3×10^{-7} bar/min (the pressure changed from 7.6×10^{-7} bar to 9.9×10^{-7} bar within 60 s). The usual pressure in the vacuum chamber is in the order of 1×10^{-7} bar to 1×10^{-6} bar.

The trap – temperature sensor

The temperature sensor inside the trap broke in half on 08/12/2020 when the trap was moved from position 6500 back to 0. Due to the breaking, JUMO 1 – trap temperature – was showing 3×10^{37} °C. This "error" value means "invalid input value" according to the manual, i.e. there is too much of resistance (broken wire). The platinum temperature sensor was replaced and soldered on 14/12/2020 by Andreas Schneider from LPC (D-CHAB).

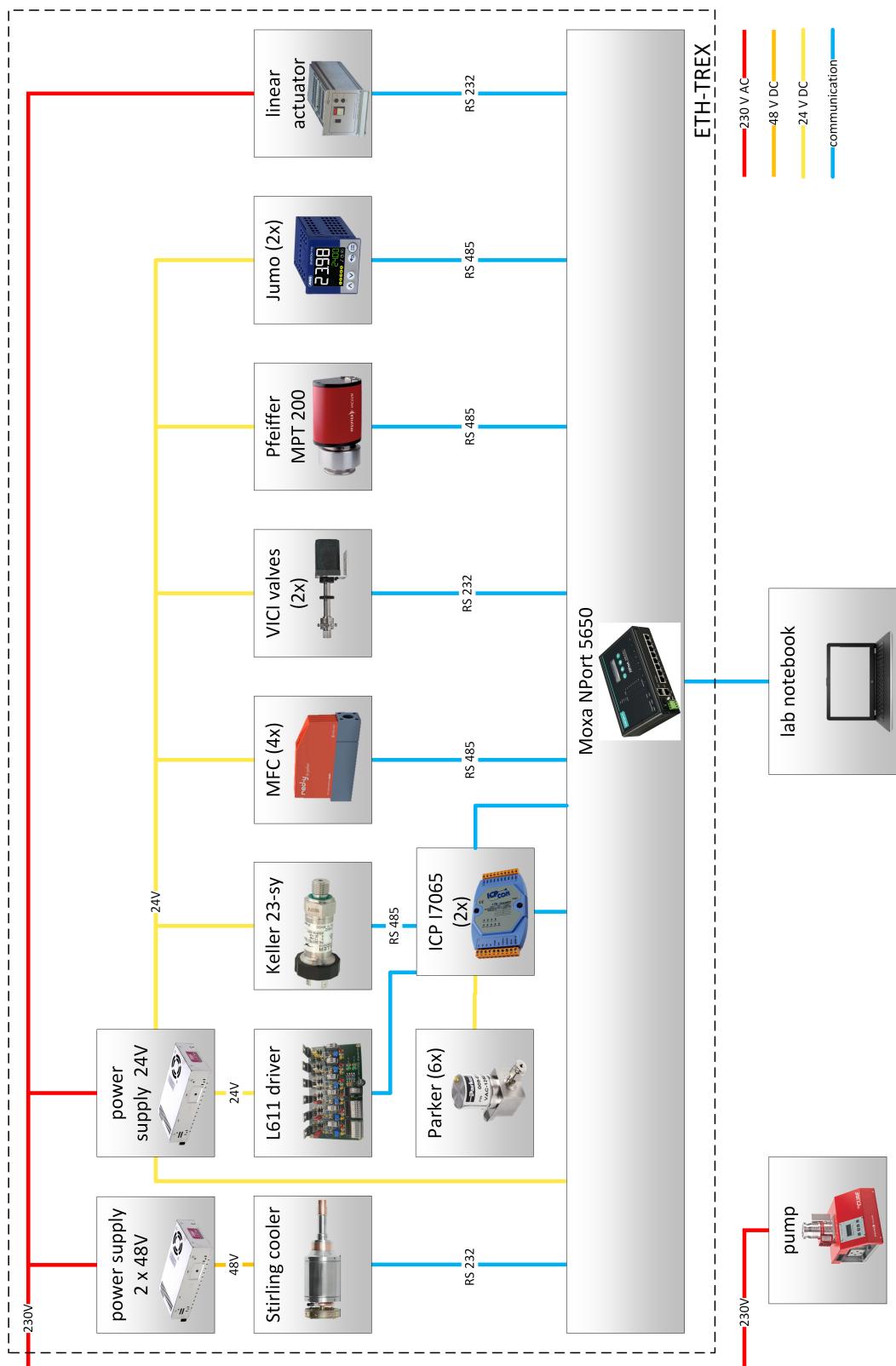


Figure 2.8: A wiring diagram of the TREX unit.

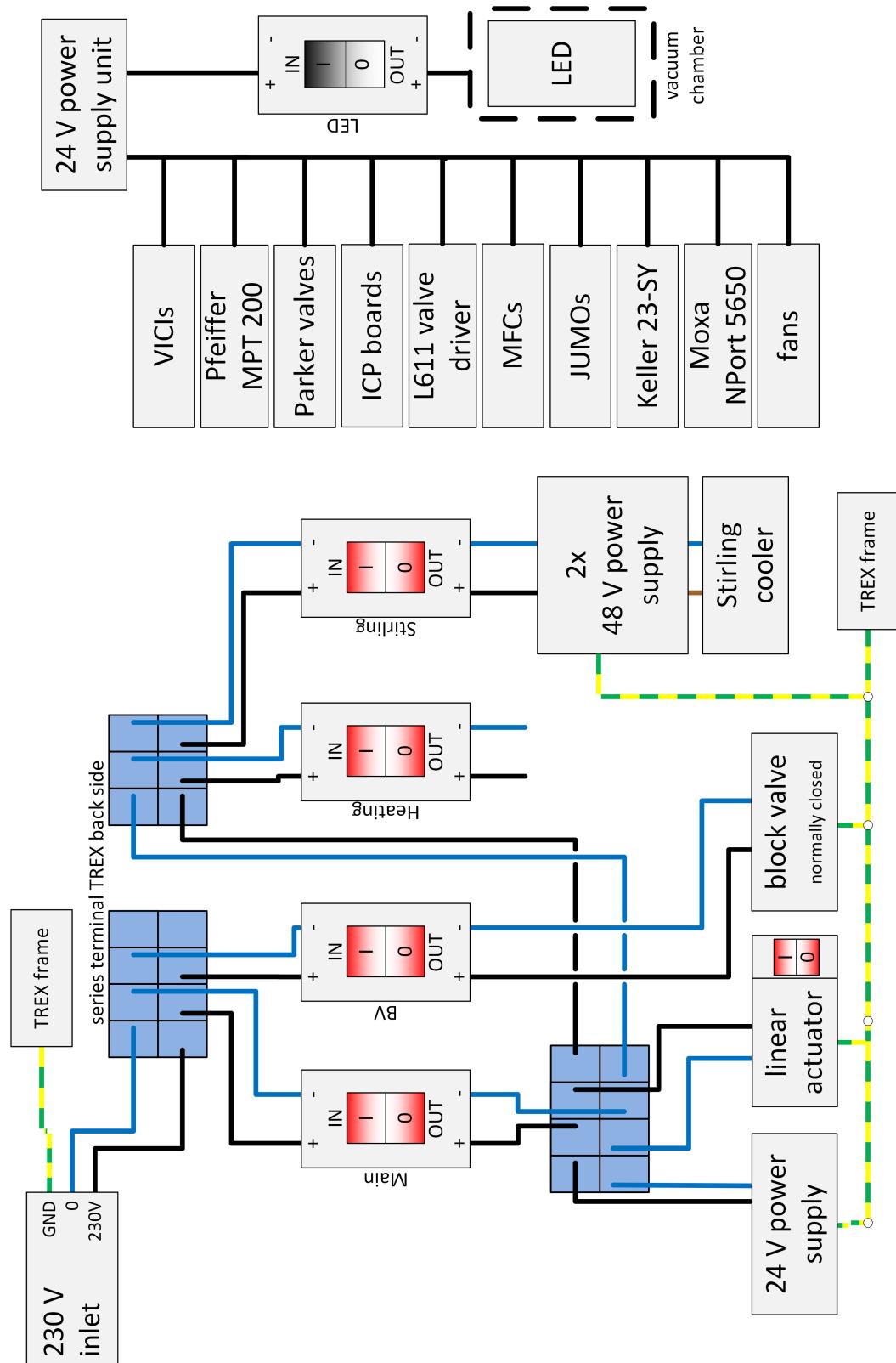


Figure 2.9: A wiring diagram of the power supply units. The red switches are the switches on the TREX front panel. Erkan left this diagram incomplete so I had to finish it. It was not possible to check every connection to the units as the TREX was on and running.



Figure 2.10: A scheme of the electrical feed-through, the view should be from the outside of the vacuum chamber. Inside the vacuum chamber, the denomination of the pins is mirrored.

The trap – breakthrough experiments

The capacity of the trap for N₂O adsorption was tested on 12/10/2021. As sample, the N₂O gas in the Messer cylinder D131733 was used. The approximate concentration of this gas is 3 ppm. The cylinder pressure was approx. 30 bar before the experiment and the cylinder volume is 50 L, therefore there was approx. 1500 L of gas. The whole cylinder had approx. 180 µmol of N₂O. The sample outlet of TREX was connected by a 1/16 inch-tube to the QCLAS-2016 instrument (Fig. 2.11). The QCLAS outlet was left as it is, connected to QCLAS-2014.

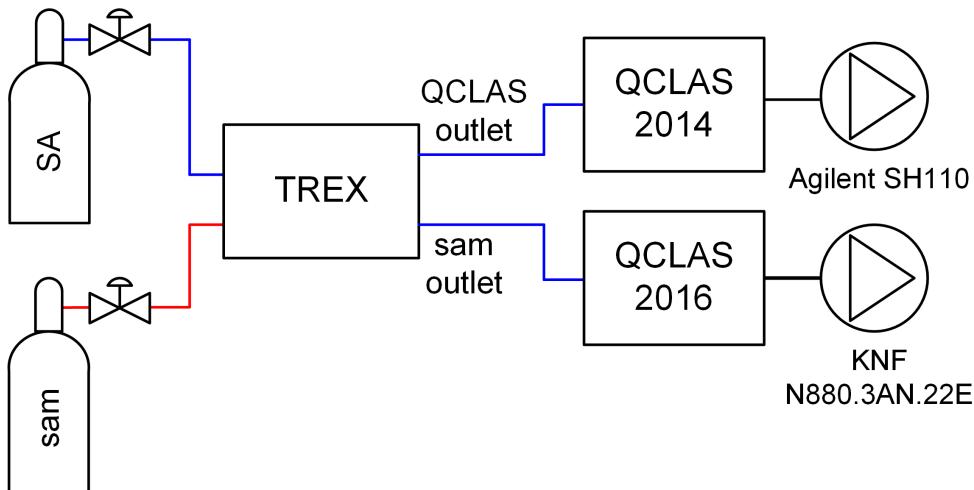


Figure 2.11: A scheme of the breakthrough experiment performed on 12/10/2021.

The QCLAS-2016 cell was cleaned prior to the experiment by purging it with SA. The used experiment file is 20211012_breakthrough_expl.xml. The adsorption phase of the preconcentration routine was extended and the rest of the routines was deleted. The sample flow during the adsorption was kept at 550 mL/min. The trap temperature was –134 °C at the beginning of the adsorption and decreased to –145.5 °C throughout the experiment. On QCLAS-2016, the pressure lock was activated to 15 Torr, which caused regular short-term disturbances in the sample flow (Fig. 2.12).

The adsorption phase lasted 3.5 h and no breakthrough was observed in this time frame (breakthrough = capacity of the trap is reached, N₂O in the sample gas is not adsorbed anymore and the concentration in QCLAS would increase from zero to 4 ppm in this case). At the end, the sample flow was stopped and the trap was flushed through by SA to QCLAS-2016 while still low in temperature (MFC 3 – 100 mL/min, VICI 1 – pos. 1, VICI 2 – pos. 12, flowing through MFC 4 and the sam outlet). Then, the

trap was detached from the baseplate and heated to 0 °C (the SA flow was kept constant). Figure 2.12 shows the sudden desorption of preconcentrated N₂O. Afterwards, the trap was heated out to 35 °C and purged for few minutes.

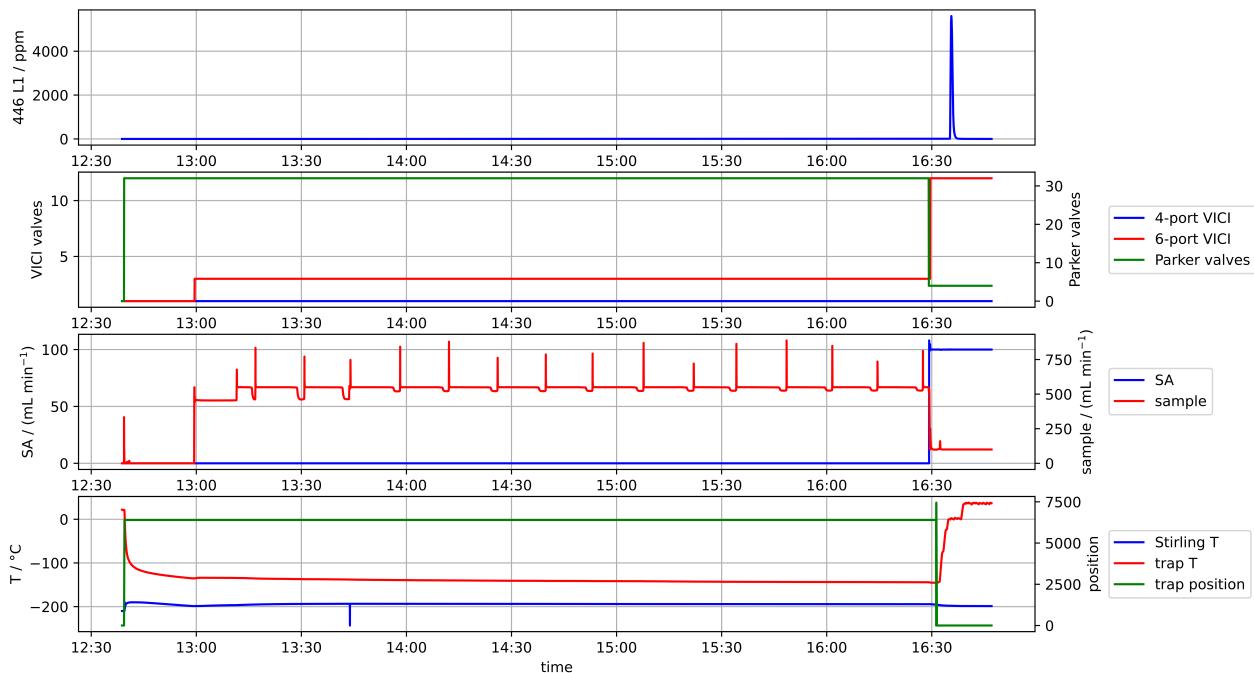


Figure 2.12: A graph for the breakthrough experiment performed on 12/10/2021.

Based on the approximate concentration of the sample gas, approx. 14.15 µmol of N₂O was trapped. This corresponds to 113.4 ± 1.5 L of the sample gas. The average trap temperature during the adsorption phase was -140.6 °C and the average sample pressure was 5.6 bar.

In breakthrough experiments of Erkan Ibraim Ibraim et al., 2017, approx. 6.8 µmol of N₂O was adsorbed (> 500 L of pressurized ambient air at 329 ppb N₂O) and no breakthrough was observed.

In breakthrough experiments performed at Empa Dübendorf together with Sarah Eggleston (October 2017), the sample gas was chosen such that it has high concentration of N₂O and the breakthrough can be observed in a reasonable time frame. In this experiment, approx. 8.2 mmol of N₂O was adsorbed in the trap before the breakthrough (2170 L of gas at 90.9 ppm N₂O; 45.3 h with gas flow of 800 mL).

The results of the experiments performed with this TREX unit are satisfactory. The adsorbed N₂O amount is well above the amount observed by Erkan, therefore the capacity of the trap is large enough for common samples.

2.1.3 Making the HayeSep D trap

June 2021 – there are these spare components: a filled trap, a stand-off, a heat pad, a wave spring, and a platinum-ceramic temperature sensor in the lab B32, in the cabinet closest to the window, the last drawer

The trap consists of a stainless-steel tube coiled 3.5 times around an aluminium stand-off (diameter 80 mm, **where it was produced? Empa workshop?**) and filled with the HayeSep D adsorbent, see Fig. 2.13. The making of the trap requires very careful handling of the materials and tools:

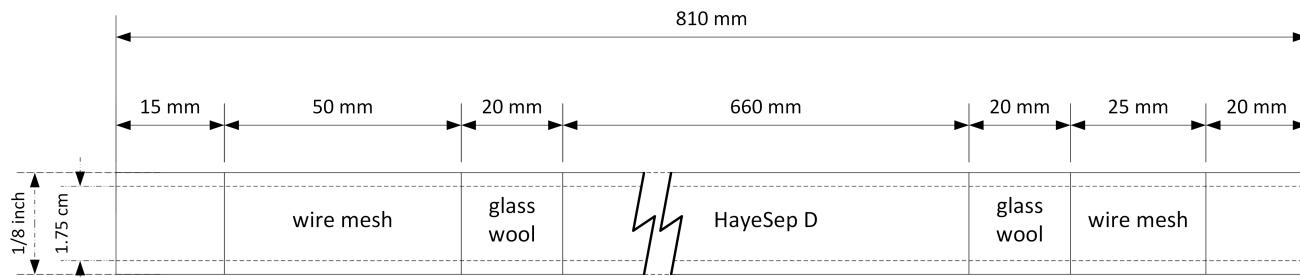


Figure 2.13: The HayeSep D trap – the content and the dimensions. The picture was made by Andrin Moosmann at Empa, modified by Kristýna for the ETH-TREX.

- a stainless-steel tube, OD 1/8 inch, tube wall 0.028 inch, total length 81 cm (Swagelok order number SS-T2-S-028-6ME for 6 m of the tube)
- HayeSep D adsorbent, approx. 2 g
- acetone, HPLC grade, 250 mL
- wire mesh
- glass wool
- vinyl gloves
- a heat gun
- tweezers
- pliers
- tape
- a Petri dish
- a Pasteur pipette
- two metal rods, diameter approx. 1.5 cm to fit inside the stainless-steel tube, length #1 15 mm, #2 725 mm or more with marks of 20 mm, 45 mm, 65 mm, and 725 mm.
- a ruler
- a pump
- an impinger
- silicone tubing
- a holder for the tube

Instructions for filling the tube

1. Clean the tube from the inner side with acetone and glass wool. Dry afterwards with the heat gun.
2. Cut two pieces of the wire mesh: 50 x 50 mm and 25 x 50 mm.
3. Roll the bigger wire mesh and insert it into the tube using the 15 mm-metal rod.
4. On the tube, mark the depth at which the wire mesh is.
5. Put the 15 mm-rod back inside the tube.
6. Tape the end of the tube with the rod inside.
7. From the other side, put inside some glass wool in small steps using tweezers and the second metal rod until the rod is levelled with the tube end at the 725 mm-mark (20 mm of the glass wool in total in the tube).
8. On the tube, mark the depth at which the glass wool is.
9. Fix the tube vertically with the open side up.
10. Attach the tube through the impinger to the vacuum pump. Keep the tape on, the rod needs to keep the position of the tube content.
11. Weigh approx. 2 g of HayeSep D into the Petri dish and mix with approx. double the amount of acetone to give a slurry.
12. Insert the slurry with the pipette into the tube and let acetone get sucked out by the vacuum pump into the impinger. Use the rod occasionally to make sure that the individual layers are compressed well.
13. Fill the tube with HayeSep D until there is 65 mm left. The total amount of HayeSep D in the trap is not that critical, the correct filling is more important.
14. On the tube, mark the depth at which HayeSep D is.
15. Seal HayeSep D with the second layer of glass wool until the rod is levelled with the tube end at the 45 mm-mark.
16. On the tube, mark the depth at which the second glass wool layer is.
17. Roll the shorter wire mesh from the shorter end and insert it into the tube using the rod until it is levelled with the tube end at the 20 mm-mark.
18. On the tube, mark the depth at which the second wire mesh is.
19. Mark also the inlet (50 mm-wire mesh) and the outlet side of the tube (25 mm-wire mesh).
20. Bake the tube overnight at 160 °C with He or N₂ flow of 0.2 Lmin⁻¹.

Instructions for making the trap

The trap was made using a stand-off dummy that is at Empa but it can be made anew if needed. The diameter of the dummy is smaller to compensate for radial springback of the tube. Materials and tools used for the trap making are following:

- a filled trap tube from the previous subsection
- a stand-off dummy (an aluminium stand-off, diameter 70 mm; piece of wood 4 x 4 x 2 cm; a steel bar 15 x 2.5 x 0.4 cm, bent into an L-shape; M6 x 25 screw, nut, and washer)
- an adjustable wrench
- a hammer
- a bench vice
- a drilling machine with a drill, diameter 6 mm
- cut-resistant gloves for mechanical protection (the edges of the stand-off threads are sharp)

If the stand-off dummy is not available, put it together following these steps and Fig. 2.14:

1. Drill a 6 mm-hole into the middle of the stand-off, into the middle of the piece of wood and into one end of the metal bar.
2. Bend the metal bar 36 mm from the center of the hole using the hammer and the vice.
3. Put the M6 screw through all components and fix them together.

Instructions to bend the tube onto the stand-off dummy:

1. Hitch the stand-off dummy into the vice as in Fig. 2.14.
2. Make two marks on the tube, 7 cm from each end. This is where the bending will start.
3. Put the end of the tube between the metal bar and the stand-off into the upper thread such that the longer part is in the upward direction of the thread.
4. Start bending the tube into the thread by grabbing firmly the stand-off and the shorter end of the tube and rotating the metal bar.
5. After one revolution, the metal bar can be removed from the dummy and the rest can be bent by hand (Fig. 2.15).
6. Stop bending when the needed length is achieved.
7. Remove the tube from the dummy. The diameter of the tube should be between 70 mm and 80 mm.

Instructions to put the tube onto the stand-off and the needed material and tools:

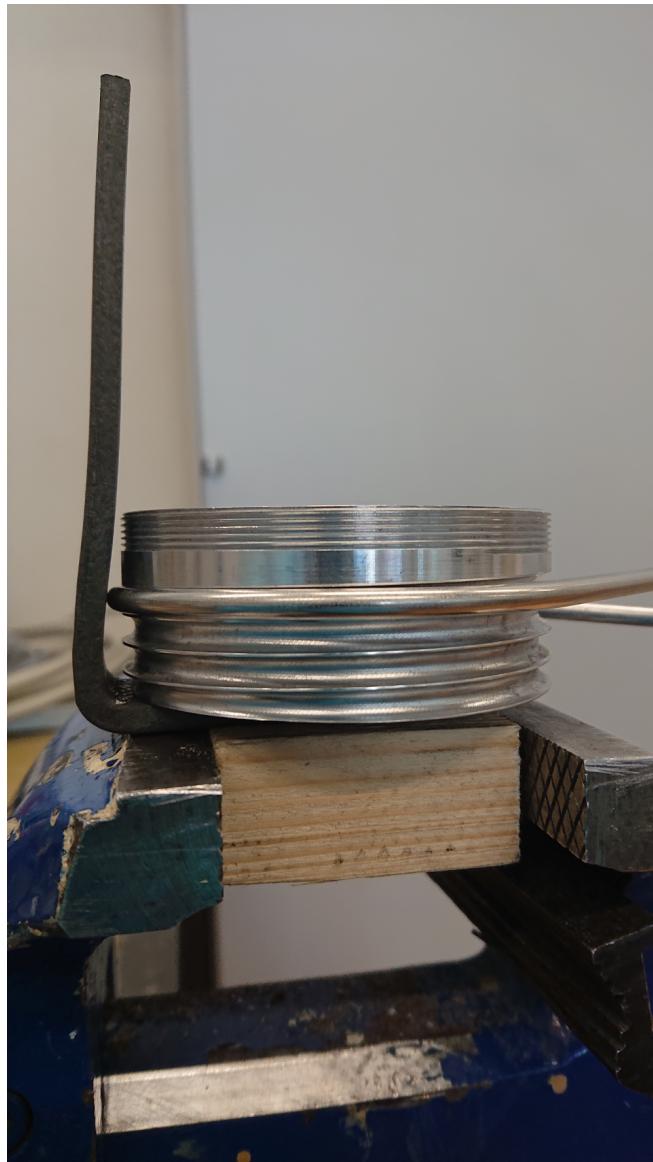


Figure 2.14: A photo of the stand-off dummy. The photo was made by Andrin Moosmann.

- a new stand-off (aluminium, diameter 80 mm)
- heat-conducting paste (Dow Corning, available e.g. at Distrelec, PN 175-59-883; Fig. 2.16)
- vinyl gloves
- cut-resistant gloves
- a platinum-ceramic temperature sensor (JUMO Mess- und Regeltechnik AG, Pt-100, PN 00038276; Fig. 2.16)
- a heat pad
- a PEEK plate
- a stainless-steel wave spring (Durovis AG, PN WF-8941-SS; 50.8 x 40.6 x 15.9 mm; the spring is heat-treated (stainless steel 1.4568) otherwise there may be problems later at lower temperature if other material is used)



Figure 2.15: Photos of the stand-off dummy from the top during bending of the trap tube (left) and the bent tube (right). The photos were made by Andrin Moosmann.

1. Apply the heat paste to the threads of the 80 mm-stand-off and the inner side of the bent tube (wear the gloves).
2. Carefully mount the tube onto the stand-off. If the tube gets stuck at some point, you need to grab the tube around all curves from two sides and move the whole thing together. It may be better to change at this point the vinyl gloves for the cut-resistant ones since there is quite a high risk of cutting yourself.
3. Connect the Swagelok 1/8 inch-female nuts to the tube.
4. Polish both flat surfaces of the stand-off with a piece of tissue dipped in distilled water or ethanol.
5. Do the same with the copper base plate of the Stirling cooler.
6. Apply a thin layer of the heat paste onto the base plate and the stand-off outer and inner surfaces.
7. Put the heat pad into the stand-off.
8. Cover the temperature sensor with a bit of the heat paste and insert it into the hole in the stand-off (Fig. 2.17).



Figure 2.16: Photos of the heat paste (left) and the temperature sensor (right).

9. Insert the wave spring to cover the heat pad (Fig. 2.17) and then the PEEK plate. The wires need to go through the cut-out space in the plate.
10. Screw the stand-off into the plastic thread on the actuator by rotating the plastic part, not the stand-off (careful with the wires here, they need to go through the space between the PEEK plate and the plastic thread). Two photos how the trap looks like when it is mounted onto the actuator are in Fig. 2.18.

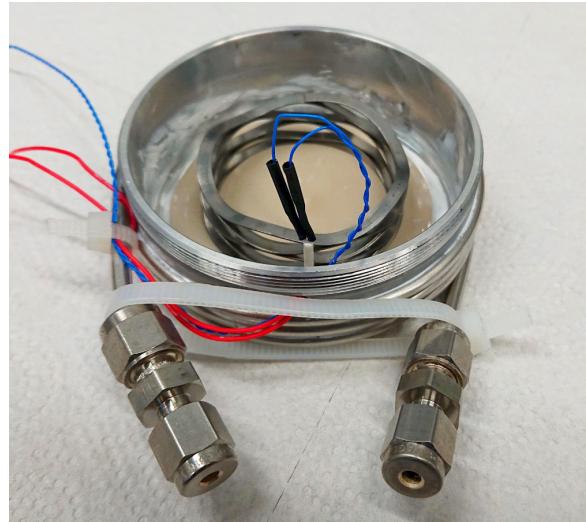


Figure 2.17: Photos of the hole in the stand-off for the temperature sensor (left) and the wave spring in the stand-off (right; the photo was made by Andrin Moosmann; in this design, the sensor wires are led out of the inner space differently, through a hole in the stand-off body).

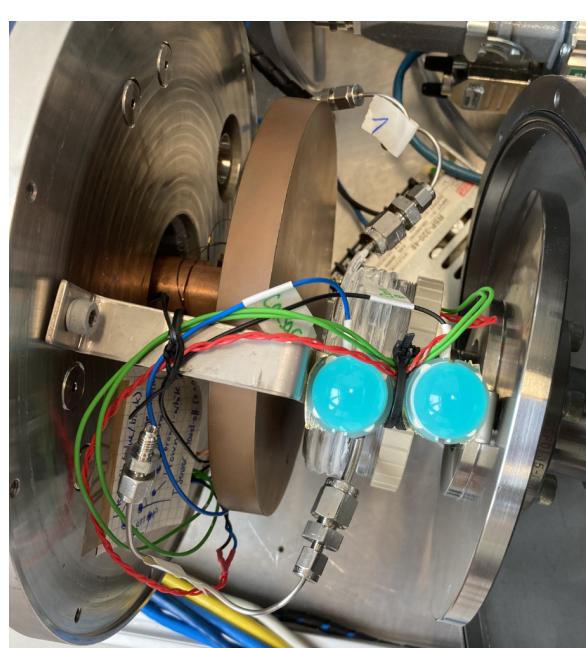
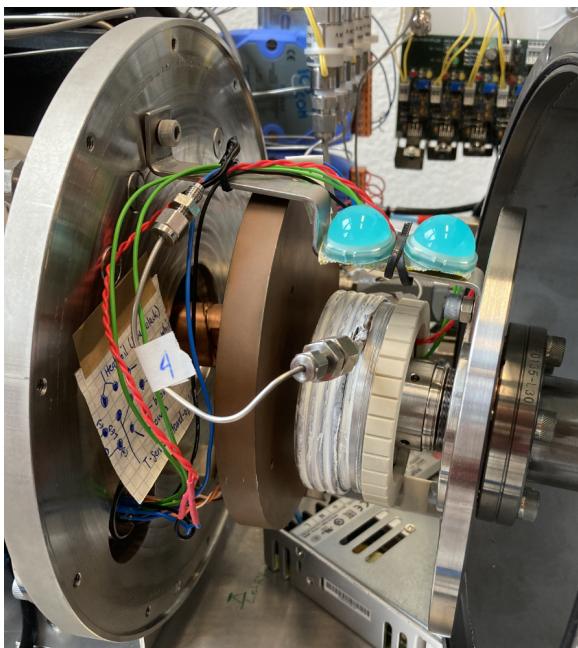


Figure 2.18: Photos of the trap mounted onto the linear actuator, the stainless-steel tubing was not yet pulled through the vacuum chamber plate. The big blue LEDs broke and were replaced in December 2020 by a set of smaller ones.

2.2 TrexOS

The software that operates the TREX unit is written in LabVIEW and is called "TrexOS" ("TREX Operating Software"). On Fig. 2.19, you can see the user interface (UI) of the TrexOS software. On the top panel, there are five cards: Main control, Control, Vars (N/A), Dilution/File readout (N/A), and Tasks. However, only the first two cards are used.

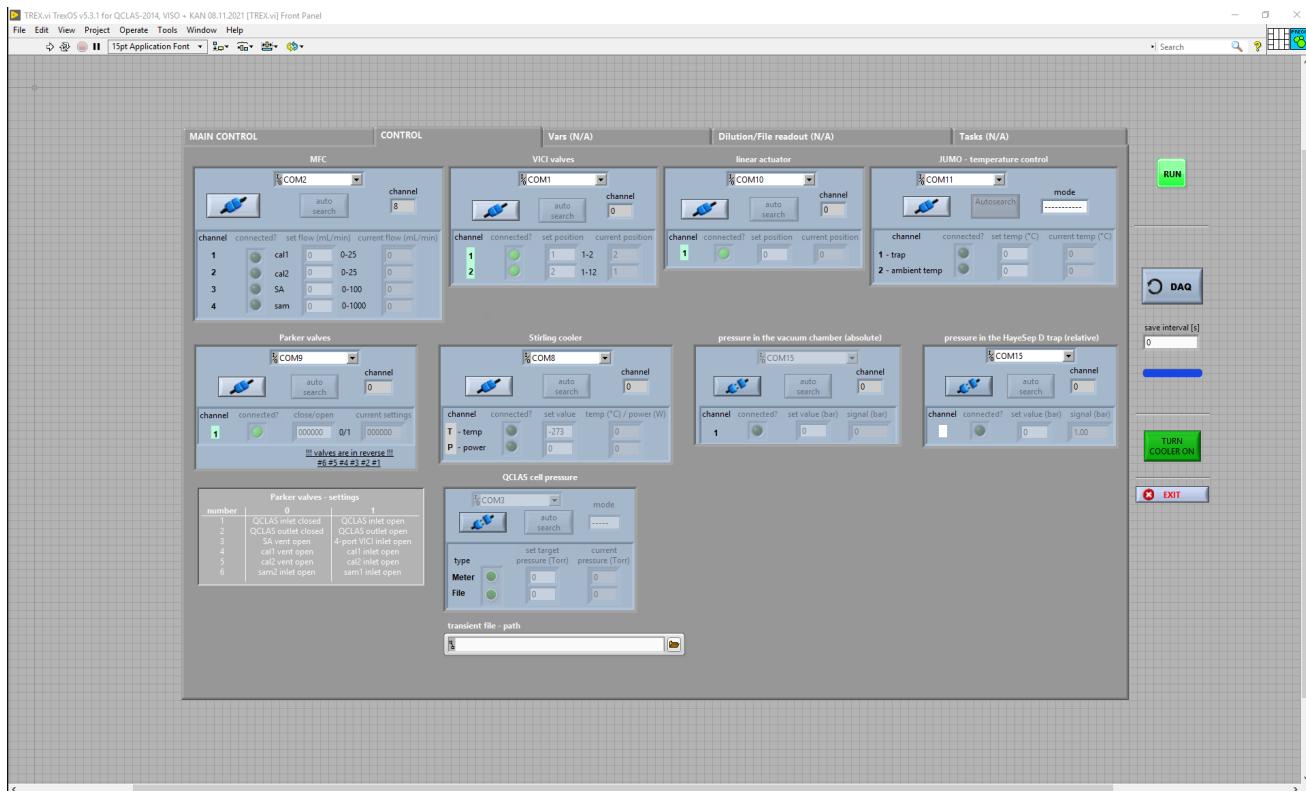


Figure 2.19: UI of TrexOS before running.

2.2.1 The Control card

On the Control card (Fig. 2.19 and Fig. 4.5), the individual components in TREX can be manually operated:

- MFC – a window to operate the MFCs, to set desired flow rates and see current values.
channel 1 – calibration gas 1, 0 – 25 mL/min
channel 2 – calibration gas 2, 0 – 25 mL/min
channel 3 – synthetic air, 0 – 100 mL/min
channel 4 – sample, 0 – 1000 mL/min
- VICI valves – a window to operate the two VICI valves, to set desired position and see current values.
channel 1 – the 2-position 4-port VICI valve
channel 2 – the multiposition 6-port VICI valve, set to the total of 12 positions
- linear actuator – a window to operate the linear actuator, to set desired position and see the current one.
channel 1 – the position, 0 – 6400

- JUMO – temperature control – a window to operate the JUMO unit on channel 1 and see current values.
channel 1 – the trap temperature, the trap can be actively heated, or the heating can be switched off completely
channel 2 – the ambient temperature, measured inside the TREX unit
- Parker valves – a window to operate the Parker valves. Under the window, there is a table showing what each Parker valve does while open or closed.
channel 1 – six Parker valves, ordered in reverse; e.g. 100000 means that Parker valve #6 is open, while 000001 means that Parker valve #1 is open
- Stirling cooler – a window to operate the Stirling cooler, to set the desired temperature and see current values.
channel 1 – the base-plate temperature
channel 2 – the power of the Stirling engine, the value can be set max. to approx. 230 W
- pressure in the vacuum chamber (absolute) – a window to show the value of the pressure inside the vacuum chamber on channel 1; in bar
- pressure in the HayeSep D trap (relative) – a window to show the value of the pressure in the adsorption trap; in bar
- QCLAS cell pressure – a window to show the cell pressure; only when the data saving in TDLWintel is active and the file path is correctly set to the transient file; in Torr
channel 1 – not active, only if there was a cable connecting the Baratron pressure meter directly with the lab notebook
channel 2 – data taken from the transient file on the QCLAS computer

2.2.2 The Main Control card

On the Main Control card (Fig. 2.20), you can start and modify the automatic course of an experiment and there is an overview of the actual progress. The fields in the upper right part can be used to quickly modify some of the parameters without going to the Control card (Set Flow, Set Vici, Set Temp, and Set Valves).

Routine manager

The Routine manager serves for loading and editing experiment database files that are saved in the *.xml format. TrexOS then goes through the file step by step, and the next command is performed only if the execution of the previous command is completed.

- On the Main Control card, open the Routine manager (the icon with two gearwheels, Fig. 2.20).
- Select an experiment database file (*.xml) in the upper left path window (Fig. 2.21).
- Underneath the path window, there are three columns – 1) routines included in an experiment (e.g. ResetALL to reset all component settings before the experiment starts, or A_S1 to perform first adsorption of sample 1 onto the trap and then desorption into the QCLAS cell); 2) procedures in a routine (e.g. DesorbSam to desorb the preconcentrated N₂O from the trap and transfer it into the QCLAS cell); 3) steps in a procedure (e.g. MFC Write for changing the gas flow through certain MFC; for complete overview of all steps, see 2.2.3).

- If you need to do modifications in the experiment file, click on the bottom middle edit database file button.
- In the next window (Fig. 2.22), select the routine that you want to modify in the upper middle menu Routine displayed and the desired procedure in the upper right menu Procedure displayed. You can change the timing, channels, and values in the right column.
- If a new routine, procedure, or step is required or if you want to delete something, use the buttons Create new, Duplicate, and Delete below the respective column.
- When the modifications are finished, save the new file by pressing the upper right Save button and then the bottom right Return button once to see the experiment overview or twice to exit the Routine manager completely (never by the X in the upper right corner, otherwise the window keeps hanging somewhere, it cannot be opened again and the whole software needs to be restarted).



Figure 2.20: A TrexOS printscrean showing the Main control card where the Routine manager and the graphical overview are located.

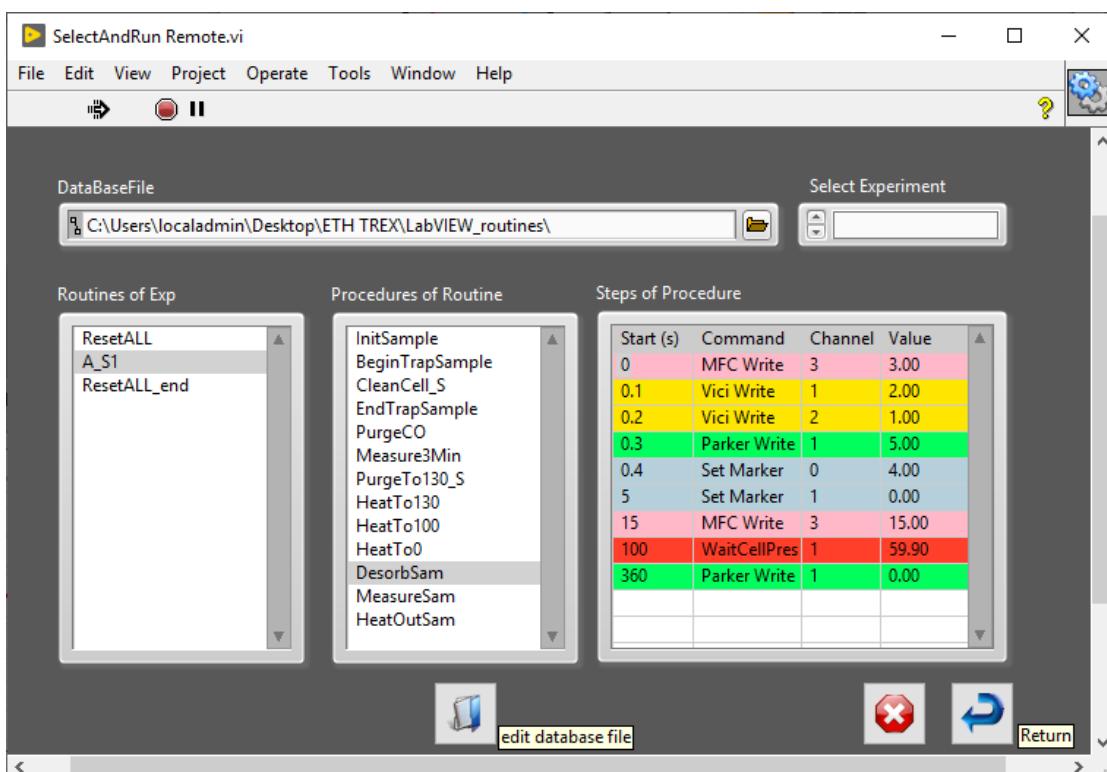


Figure 2.21: The Routine manager window where the experiment database file can be loaded and the individual routines, procedures, and steps can be seen.

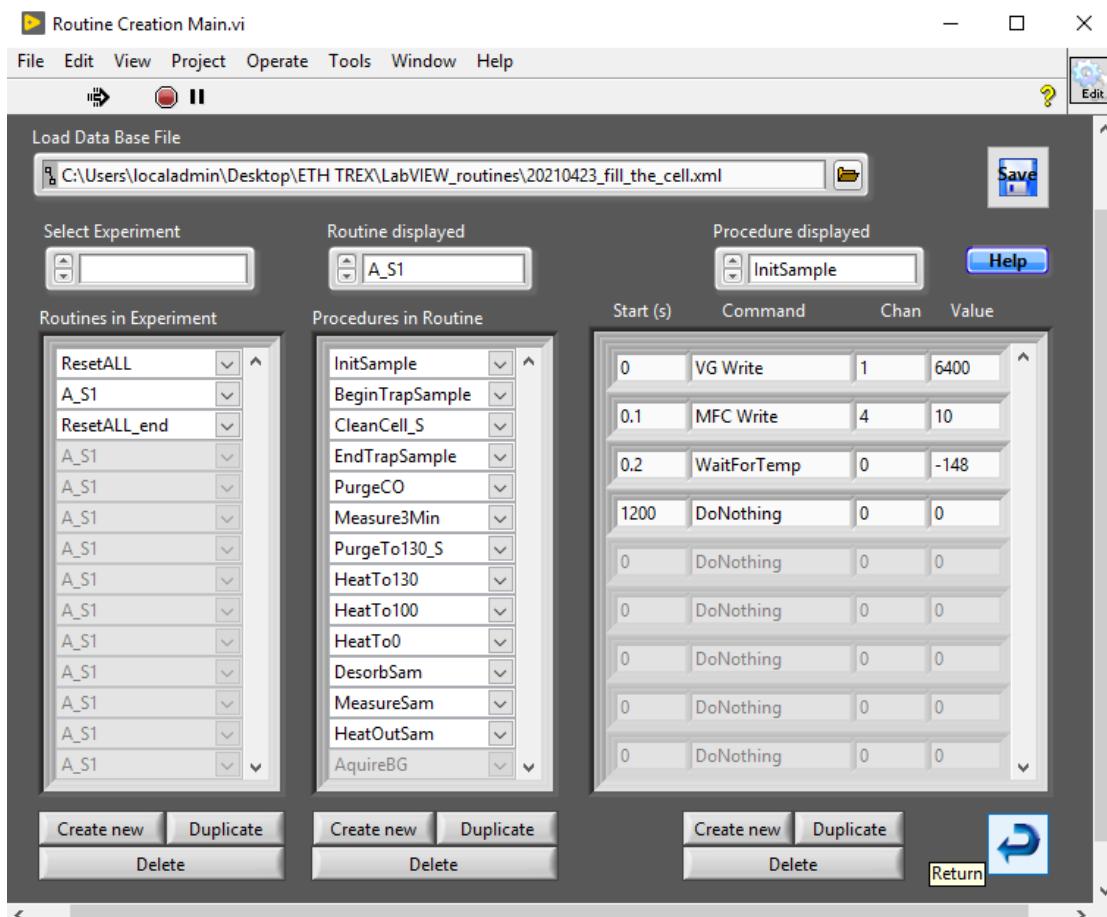


Figure 2.22: A part of the Routine manager where the experiment database file can be edited and saved.

2.2.3 Steps in a procedure

Steps in a procedure are programmed using the following set of commands. Each step is set to a starting time in seconds, a command name, a channel, and a set value. Steps that are supposed to be executed at the exactly same time should have in between a 0.1 s break otherwise some of the commands may not actually make it through the communication channel.

- Cryotel Write – to control the Stirling cooler; channel 1 – temperature (-210°C), channel 2 – power. The power is generally not controlled here, its value depends on the difference between the actual and the set temperature.
- Do Nothing – this is the default command. It literally does nothing. It can be used for setting a time limit until execution of a next command.
- Jumo Write – to set temperature of the JUMO temperature controller; channel 1 – the trap, channel 2 – the ambient temperature. JUMO 2 does not control actively any temperature, it only measures the temperature inside the TREX box. However, the set value on channel 2 has to be greater than that on channel 1, otherwise the heating of the trap, which is controlled by JUMO on channel 1, does not switch on (most probably caused by some weird cross-communication between the two channels). Therefore, the value of channel 2 is set to 35°C or 45°C .
- MFC Write – to set the gas flow through an MFC; channel 1 – cal1, channel 2 – cal2, channel 3 – synthetic air, channel 4 – sample.
- Parker Write – to either open or close a Parker valve; 0 means closed, 1 means open. All six Parker valves are on channel 1. Figure 2.23 has a table from TrexOS showing what each Parker valve does while open or closed. The valves are ordered in reverse (e.g. 100000 means that Parker valve #6 is open, while 000001 means that Parker valve #1 is open), and the same applies also in the Routine manager. In addition, in Routine manager the six-digit number, describing the required Parker valve configuration, is treated as if written in the binary system and therefore has to be entered after conversion to the decimal system, e.g. 100000 has to be written as 32 and 000001 has to be written as 1. Use e.g. [this site](#) to convert from binary to decimal.
- Set Marker – to set a marker value based on the type of gas currently being processed, channel is always set to 0. The marker can be used later for data analysis.
- VG Write – to set a position of the linear actuator on channel 1; 0 – the default position, 5000 – the intermediate position for purging CO out of the trap, 6400 – the trapping position when the trap is fully pressed against the base plate of the Stirling cooler.
- Vici Write – to set a position of the VICI valves; channel 1 – the 2-position 4-port valve, channel 2 – the multi-position 6-port valve set to the total of 12 positions.
- WaitCellPresN2O – the next command is executed once the set pressure is reached or after the time at which the next command is set has passed. There are two channels: 0 – the condition for checking the pressure value is set to "equal or lower as", for lowering the pressure; 1 – the condition is set to "equal or higher as", for increasing the pressure. **This command should be always the second last command in a procedure, otherwise the following commands get to be waiting till the time of the previous command passes!**

- WaitForTemp – **This command does not work!** Someone started implementation in TrexOS but did not finish it. However, this command is not needed so I did not work on finalizing the implementation (it is quite a complex task). In normal operation, the trap is left to cool down for certain time and in the meantime, the calibration gas is measured. After that time, it is assumed that the trap is already cold enough to start the N₂O adsorption. (In theory, the command should work similarly to WaitCellPresN2O: the next command is executed once the set temperature is reached or after the time at which the next command is set has passed. There are two channels: 0 – the condition for checking the temperature value is set to "equal or lower as", for lowering the temperature; 1 – the condition is set to "equal or higher as", for increasing the temperature.)

Parker valves - settings		
number	0	1
1	QCLAS inlet closed	QCLAS inlet open
2	QCLAS outlet closed	QCLAS outlet open
3	SA vent open	4-port VICI inlet open
4	cal1 vent open	cal1 inlet open
5	cal2 vent open	cal2 inlet open
6	sam2 inlet open	sam1 inlet open

Figure 2.23: A table from TrexOS showing what each Parker valve does while open or closed.

3 Description of QCLAS and TDLWintel

3.1 QCLAS

A commercial dual-laser spectrometer from Aerodyne Research, Inc. (Billerica, MA, USA) is used in the setup. The instrument consists of two parts (Fig. 3.1): the temperature-controlled optics module with laser sources, optics guiding the laser beam, a multi-pass cell, and detectors; and the electronics compartment with laser drivers, temperature controllers, and a computer running the operating software (described in Section 3.3). Auxiliary components include a thermoelectric chiller (OASIS), which provides cooling liquid at a constant temperature (15°C with precision of $\pm 0.05^{\circ}\text{C}$) to stabilize the temperature of the lasers, the detectors and the electronics compartment. The optics module is installed on a temperature-controlled baseplate that is isolated from the electronics by springs in order to reduce vibrations.

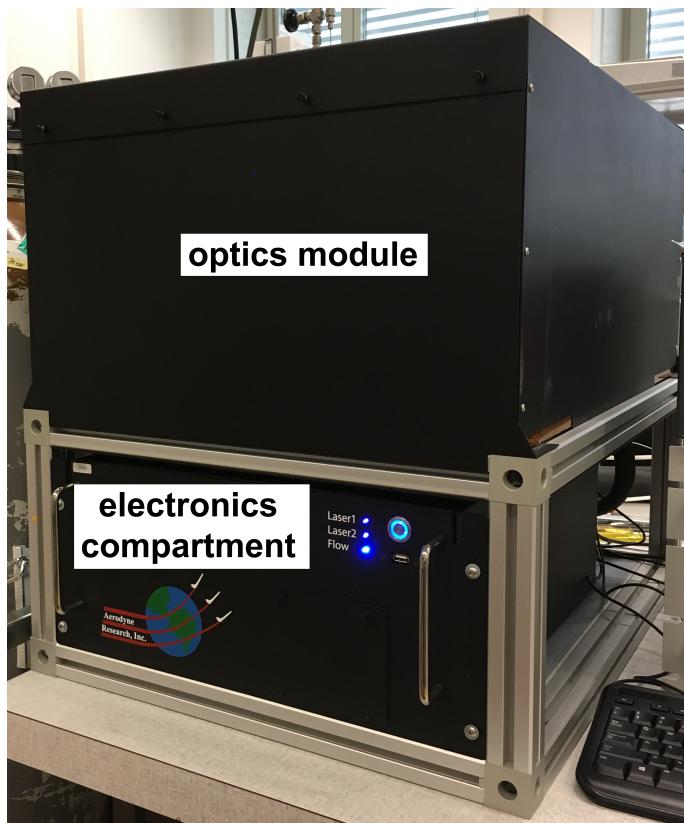


Figure 3.1: The dual-laser spectrometer with the optics module and the electronics compartment from Aerodyne Research, Inc.

The spectrometer includes two laser sources, both of which are continuous-wave (cw), room-temperature (RT), distributed feedback (DFB) quantum cascade lasers (QCL; Alpes Lasers, St-Blaise, Switzerland). The lasers are enclosed in two individual gas-tight temperature-controlled housings mounted inside the optics module (see Fig. 3.2). The characteristics of the chosen lasers are summarized in Table 3.1. A range of laser temperature is given, as it was necessary to gradually decrease the temperature over the lifetime of the lasers in order to stabilize the emission frequency as the laser active material degraded.

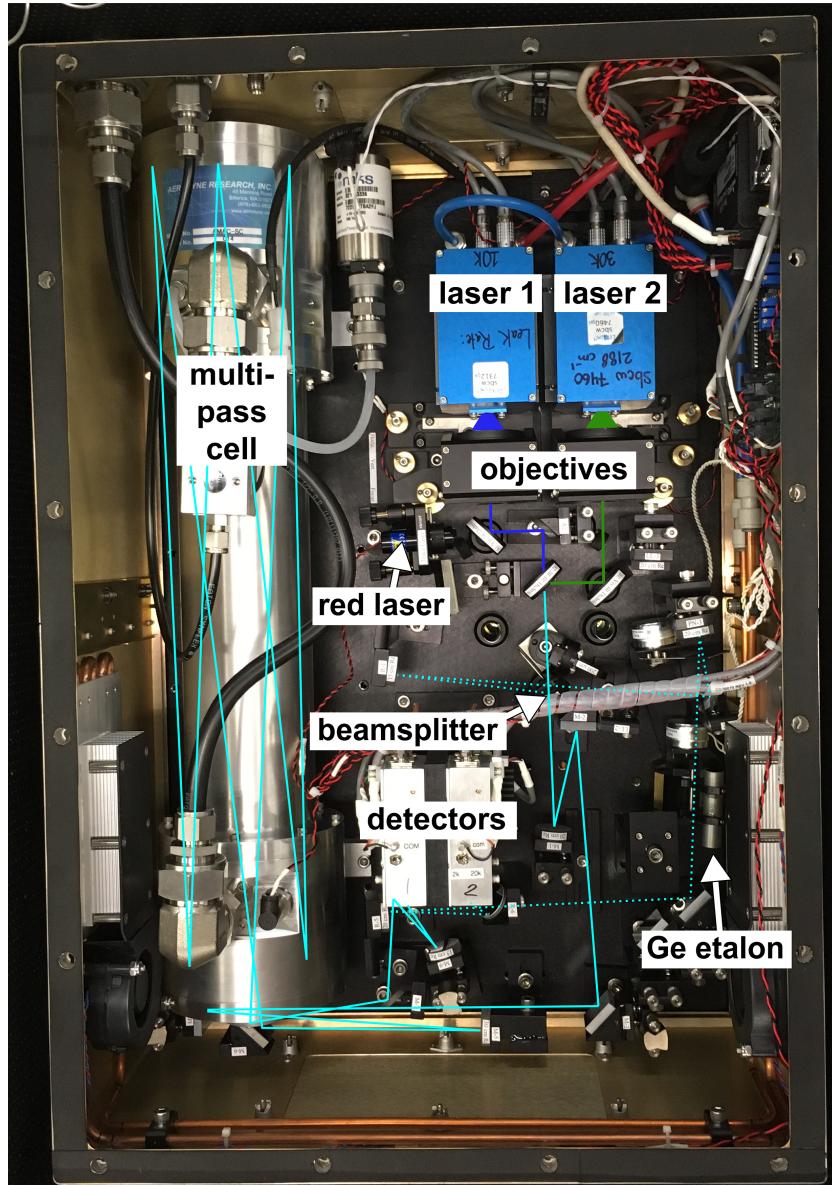


Figure 3.2: Main parts of the optics module of the spectrometer and visualization of the optical paths of laser 1 (dark blue), laser 2 (green), and combined laser beam – the main path (cyan, solid) and the reference path (cyan, dashed).

Table 3.1: Characteristics of the used quantum cascade lasers

laser	central wavenumber / cm⁻¹	T / °C	current / mA
1	2187.98	13.0 – 11.7	290
2	2195.95	(–10.3) – (–7.5)	150

At the output window of each laser housing, a reflecting objective collects and collimates the emitted diverging laser beam (Fig. 3.2). The objective of each laser is installed on a three-axis stage that can be used to adjust its focus. The individual laser beams are combined using an IR-IR combining mirror; the resulting beam is depicted by the solid cyan line in Fig. 3.2. The beam is then guided by reflective mirrors to an astigmatic multi-pass Herriott cell (Herriott et al., 1965) with a volume of approx. 2 L (the more exact volume was determined in Section 3.2). The cell body is made out of nickel-plated aluminium (the base length 47 cm) and equipped with two spherical mirrors with different radii of curvature (i.e. with a toric surface; diameter of 7.8 cm) with high-reflectivity mirror coatings (McManus et al., 2011). The leak of the QCLAS cell was determined on 01/10/2021 to be less than 0.01 Torr/min at 70 Torr. The laser beam enters and exits the cell through a central coupling hole of the same mirror.

Within the cell, the beam traverses an optical path length of approx. 200 m between the spherical mirrors (approx. 500 passes), after which it exits the cell and is collected at one of two thermoelectrically (TE) cooled detectors. The second detector is used for detection of the "reference beam" (laser light that passes only through the optics compartment, without passing through the cell), which is used for power normalization and for determination of the laser tuning rate. After the two beams coming out of the laser housings are combined on the IR-IR combining mirror, a reference beam (cyan dashed line in Fig. 3.2) is split from the original laser beam using a beamsplitter. An etalon made out of germanium (Ge) can be flipped into the optical path of this separated beam for setting up laser tuning rates using the TDLWintel software. More information on the function of the etalon is provided in Section 3.3. Close to the IR-IR combining mirror, a visible red trace laser is positioned and co-aligned with one of the QC laser beams, which allows for visualization and alignment of the whole beam.

3.2 Experiment – the QCLAS cell volume

This part describes experiments determining the volume of the QCLAS cell and the tubes leading to and from the gas inlet and outlet and the pressure meter, when the cell is isolated in the batch mode. The volume was determined in two experiments, on 01/10/2021 and 05/10/2021, based on the difference in cell pressure before and after filling the cell for 60 s with 10 mL/min and 20 mL/min of SA, respectively. In each experiment, the filling was repeated five times. The cell volume was determined to be 2359 ± 84 mL ($k = 2.57$; 01/10/2021) and 2662 ± 69 mL ($k = 2.57$; 05/10/2021). It can be then assumed that the cell volume (including the tubes and the pressure meter) is approx. 2.5 L.

3.3 TDLWintel

The spectrometer is supplied together with the TDLWintel¹ software (Nelson et al., 2002), which allows the user to control the spectrometer, i.e. drive the lasers and operate the detectors, calculate spectra, analyze the spectra to obtain mixing ratios of analyzed molecules, and save the resulting data. QCLAS instruments provide the user quite a bit of freedom, as the system can also be operated with different parameter settings, such as the selection of spectral lines for quantification, wavenumber calibration, sample flow rate, and pressure. The following section provides examples of features and specific settings of the TDLWintel software; the complete functionality is described in more detail in the software manual.

One important functionality of the software is controlling the QCL sources and setting up a frequency scan. The laser settings include the parameters in Table 3.1, as well as additional parameters for setting up a voltage ramp (see Table 3.2). The lasers are driven by a voltage ramp generated by the software in pulsed mode with a frequency of 1.4 kHz. The voltage ramp uses a total of 830 points for the frequency scanning (Fig. 3.3). Of these, 400 are used for laser 1, 350 are used for laser 2, and at the end of each frequency scan, after laser 2 has been turned off, 80 are used to determine the zero signal without any laser light at -5 V. The zero signal is needed to calculate the resulting transmission and absorption.

Table 3.2: Specifications of the voltage ramp settings of the dual-laser QCLAS

laser	ramp range / V	ramp slope / steps per channel	central value / V
1	2.9298	24	0
2	3.9521	37	5.5

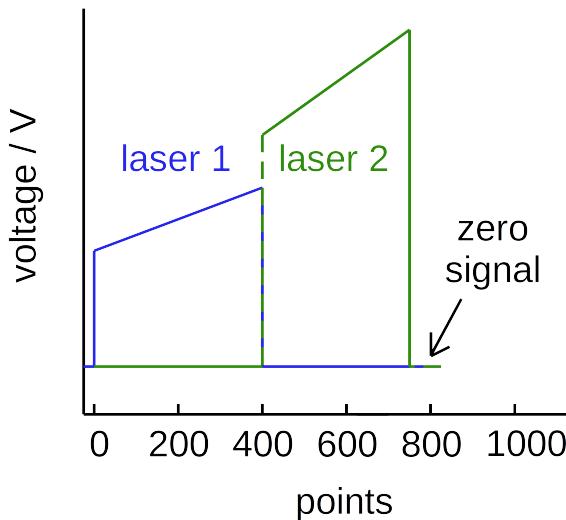


Figure 3.3: A typical voltage ramp used for driving the lasers in a dual-laser QCLAS.

¹TDL stands for Tunable Diode Laser. Wintel is a blend of words "Windows" and "Intel" that was used to designate cooperation for production of personal computers using Intel processors and Windows operating systems.

As the relationship between the applied voltage and the emitted laser frequency is nonlinear, the frequency scale has to be linearized. This should be performed regularly by switching the Ge etalon into the reference beam (cyan dashed line in Fig. 3.2) and analyzing the resulting signal with the second detector. The procedure for this is described in Section 6.2.2. The laser beam undergoes multiple reflections inside the etalon, creating a phase difference between the transmitting and reflecting beams at the exit of the etalon, thus causing a pattern of constructive and destructive interference of the beams. The transmission spectrum of the etalon has a series of peaks separated by a characteristic distance known as the free spectral range (0.024 cm^{-1} for the etalon used in this spectrometer). In the end, the measured position of the interference peaks is used to linearize the frequency scale by interpolating the positions with a cubic spline function, which is used as the laser tuning-rate function.

The user interface of the software has two main windows that are commonly used (see Fig. 3.4), but additional subwindows can also be activated to monitor the status of the instrument (not discussed here). The "Absorption Spectrum" window displays the currently measured spectral signal, i.e. the laser intensity versus the frequency scan number (the channel point). The second major feature of the software is setting up a spectral fit, i.e. interpreting spectra by fitting a model spectra. Fit markers are placed on the spectrum at particular locations to define baseline regions ([] markers in Fig. 3.4), peak positions (^ markers in Fig. 3.4), number of species in the fit, etc. Spectra are fitted in real time by the software through the application of a non-linear least squares analysis using the Levenberg-Marquardt iterative algorithm for solving non-linear equations (Levenberg, 1944; Marquardt, 1963). The baseline signal is described as a quadratic or cubic polynomial (this order is adjustable), and the absorption lines are described by the Voigt profile². The results of the fitting are shown in real time in the "Concentration Stripchart" window (in Fig. 3.4 on the right) in the form of mixing ratio in ppb for all analyzed N₂O isotopocules. The isotopocule mixing ratios are normalized to the abundance of the species 446, assuming natural isotopic composition.

Spectral fitting with the software TDLWintel requires species-specific fit parameters. The program allows the user to analyze up to 16 species. For each species, several parameters have to be set. Most importantly, the line parameters must be imported from the HITRAN database (Rothman et al., 2013), specifically the line strengths (line intensity, in $\text{cm}^{-1}/(\text{molecule} \cdot \text{cm}^{-2})$), the frequency of the absorption lines (in cm^{-1}), the air-broadened half width at ambient temperature and pressure (pressure-broadening coefficients, in $\text{cm}^{-1}/\text{atm}$), the energy of the lower transition state (in cm^{-1}), and a constant for calculation of the temperature dependence of the air-broadened half-width (Nelson et al., 2002; Rothman et al., 2013). The parameters are stored in *.hit files, which can be downloaded from the database website (hitran.org) for individual isotopocules for a specified spectral region.

For data storage, the measured signal and the mixing ratios can be saved using the control buttons "ass" (automatic spectral save) and "wd" (write to disk), respectively; these are found at the bottom left of the user interface (Fig. 3.4). Next to the control buttons, a status bar shows the laser intensity measured by the first detector, as well as the cell pressure (in Torr) and temperature, which are used as input variables for the fitting. The laser control panel to the right of the status bar serves to adjust the laser temperature that consequently shifts the laser frequency.

²The Voigt profile is commonly used in molecular spectroscopy as it can describe both the Doppler and the pressure broadening of an absorption line. Mathematically, it is described as a convolution of the Gaussian and the Lorentz profile functions that model the Doppler and the pressure broadening mechanisms, respectively (Huang et al., 2003).

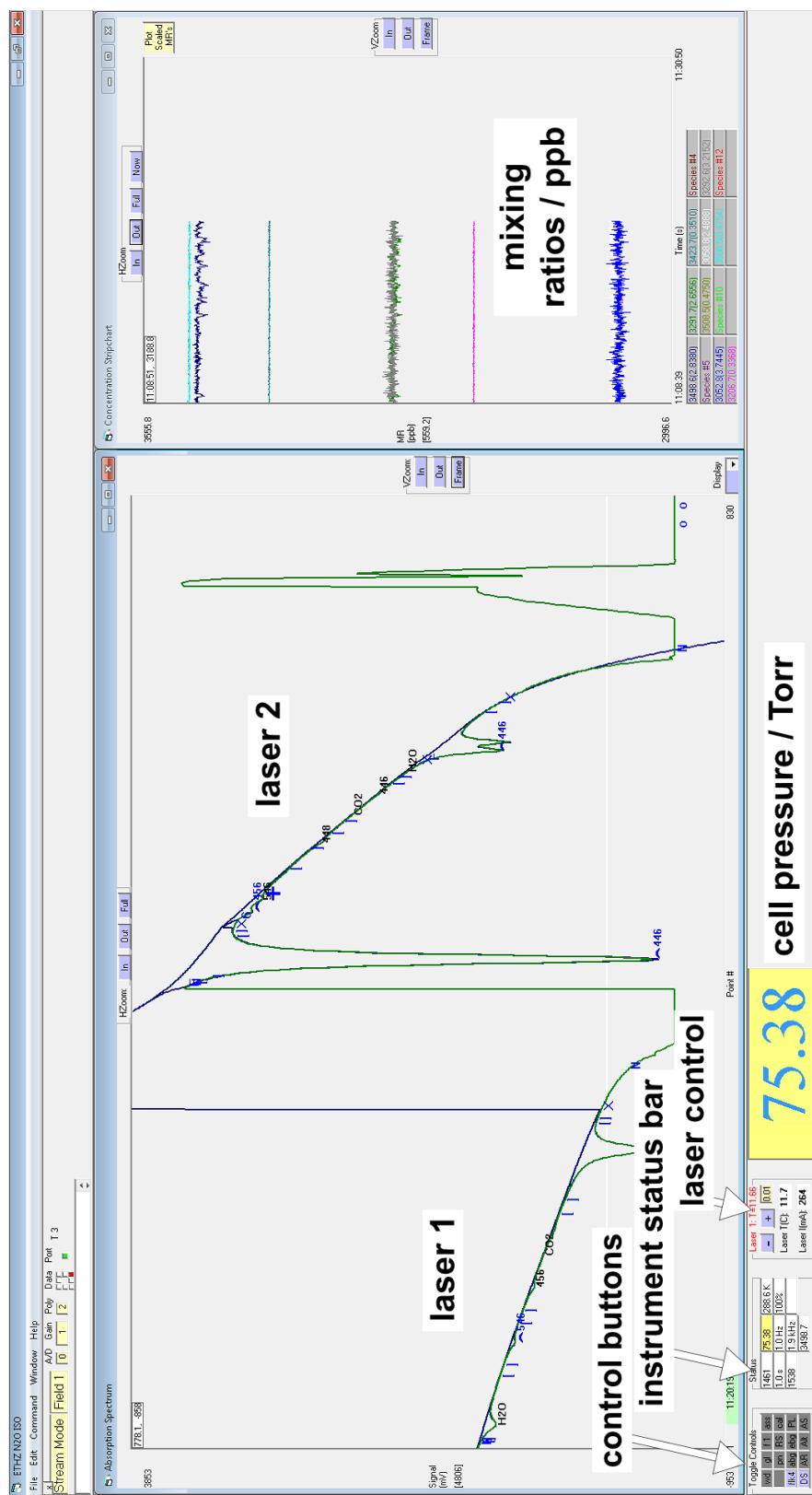


Figure 3.4: The user interface of the TDLWintel software. The important features are highlighted and described in the main text.

When the control button "wd" is on, the measurement output data are stored in two types of ASCII files. The *.str files contain the fitting results, i.e. the mixing ratios over time (1 s data). The *.stc files record conditions during the measurement, e.g. the intensity of the lasers, the cell pressure and temperature, the temperature of the lasers, and the χ^2 parameters that provide an estimate of the goodness-of-fit for the non-linear fitting procedure. The control button "ass" activates saving of spectral files *.spb and *.spe, which are crucial if the spectra need to be reanalyzed and refitted.

The spectra should be measured at rather higher pressure (e.g. 45 Torr or 70 Torr). At 20 Torr, it was observed that the fit is very unstable and it often oscillates between two levels as the peaks are too narrow and sharp. At higher pressure, this was usually not a problem.

4 Description of the TREX-QCLAS system and operation

In this chapter, first the whole TREX-QCLAS system including periphery devices is presented. Second, starting of the TREX and the QCLAS units is described. Then, instructions for operation of the TREX-QCLAS system for a measurement are summarized. At the end of this chapter, you can find a set of steps in order to shut down the whole system.

4.1 The TREX-QCLAS system

The TREX-QCLAS system comprises mainly of the TREX and the QCLAS units that were introduced in the past two chapters. However, there are other essential devices that are crucial to run the system. Figure 4.1 shows a diagram of the whole setup.

- An OASIS cooler to provide cooling liquid for the QCLAS instrument. The set temperature is 14 °C.
- An OASIS cooler to provide cooling liquid for the Stirling cooler in the TREX unit. The set temperature is 20 °C.
- A TriScroll vacuum pump serving to evacuate the QCLAS optical cell.
- A Pfeiffer HiCube turbomolecular pump serving to evacuate the TREX vacuum chamber. A block valve is attached between TREX and the pump in order to protect the pump from high-pressure strikes – however, for the automatic shutting off in case the pressure increases too fast, the block valve would need to be actually connected with a wire to the turbomolecular pump! In the current design, it is not done. So it serves just as a physical separation that can be manually switched on using a button on the TREX front panel.
- A switch box to connect both the QCLAS PC and the lab notebook to the internet and to the same network and to connect the lab notebook with the TREX and the QCLAS units. Internet connection on the QCLAS PC does not work.
- A set of gas cylinders – calibration gases (`cal`), synthetic air (`SA`), and sample (`sam`; or a sampling line).

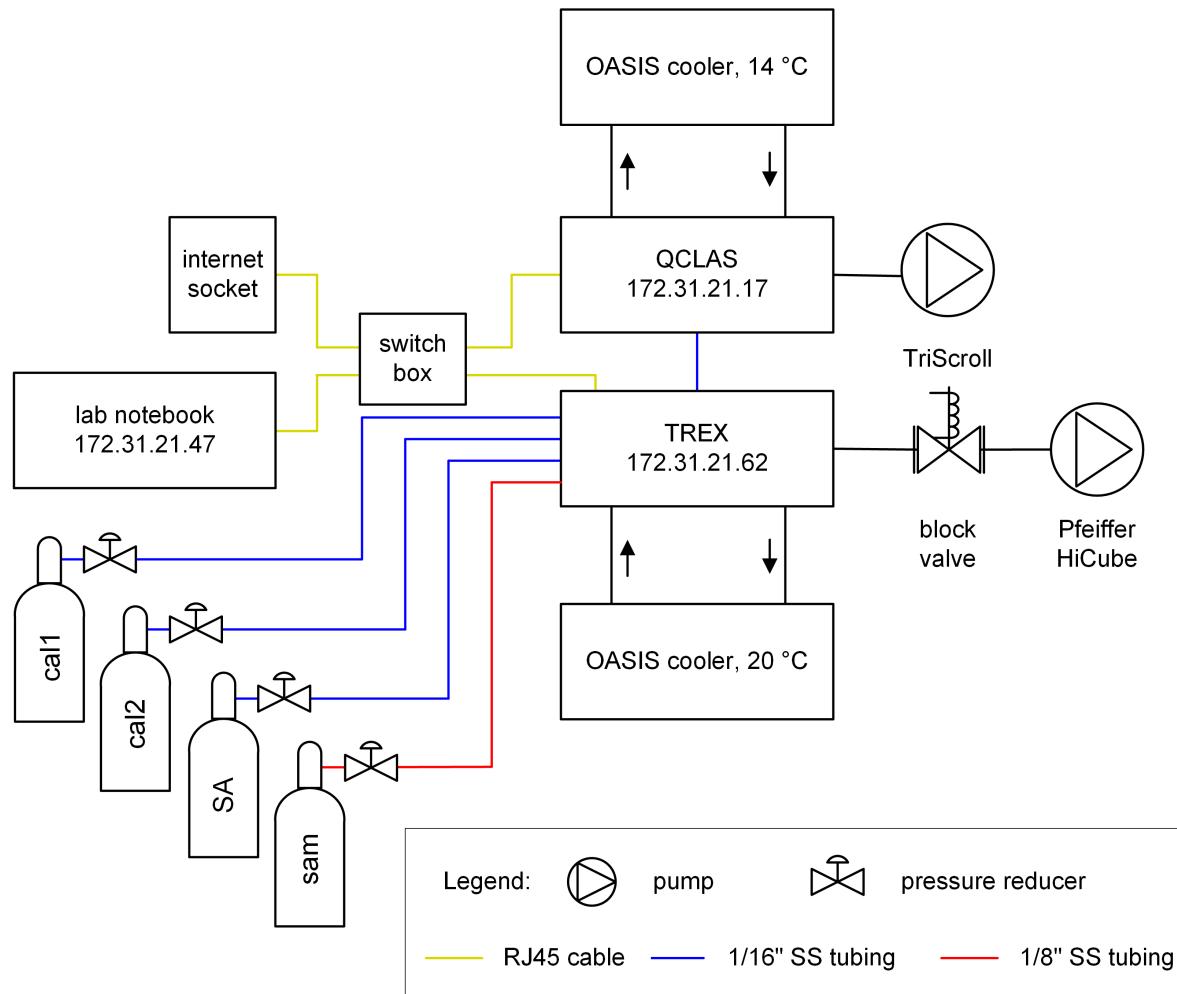


Figure 4.1: A diagram of the TREX-QCLAS system, showing set temperatures of the OASIS coolers and IP addresses of the network components.

4.2 Starting the TREX unit

In this section, steps to start up the TREX unit are described successively. Prerequisites for the start and operation of the TREX are:

- The vacuum chamber is connected to the turbomolecular pump through the block valve. Do not start the pump yet.
- The Stirling cooler is connected to the external OASIS cooling unit that is set and stabilized to 20 °C. Check if the connections are not leaking.

To start the TREX unit, the steps are following:

1. Turn on the switch on the back panel, next to the power cord.
2. On the front panel, turn on the switches Main, Heating, Cooler, and BV (block valve) and the big switch for the SimStep linear actuator.

3. Start the turbomolecular pump by pressing the switch next to the power cord and then the bottom right button under the display. The rotor frequency should increase quite quickly to 1500 Hz (on the display, item #309). Never start the pump when there is atmospheric pressure in the vacuum chamber and the block valve is still closed. If you open the block valve later and let the atmospheric pressure hit the running turbomolecular pump, the pump will be destroyed!
4. Start the notebook and log in – username: localadmin, password: abcd.1234.
5. On the Desktop, open the shortcut to the folder LabVIEW software TrexOS. There, open the most recent folder, currently ETH-TREX-20211108_Kristyna_QCLAS_2014, and the TREX.vi file.
6. On Fig. 4.4, you can see the user interface (UI) of the TrexOS software.
7. Click on Run on the left in the upper navigation bar, it is the first white arrow.
8. On the Control card, click on Run in the upper right part of the UI. The button should show now bright green Online. After pressing the button, all TREX components should automatically connect – lights under connected? for each component should turn bright green. For some components, this takes a bit more time (the slowest ones are the MFCs).
9. Check under MFC (mass flow controllers) that the current flow is 0 mL/min on all four channels.
10. Check under VICI valves that the current position is 1 on both channels.
11. Check under linear actuator that the current position is 0.
12. Check under Jumo – temperature control that the set temperature is 25 °C on channel 1 and 45 °C on channel 2. Often it says –200 °C and –100 °C, respectively, after starting TrexOS; if that happens, overwrite manually the values. Generally, the set temperature on channel 2 has to be greater than that on channel 1, otherwise the heating of the trap, which is controlled by JUMO on channel 1, does not switch on (most probably caused by some weird cross-communication between the two channels).
13. Check under Parker valves that all Parker valves are closed, i.e. current settings are 000000.
14. Check pressure in the vacuum chamber, the value should be around $2 - 3 \times 10^{-6}$ bar. If it is higher than that but decreases, wait until it drops down before going to the next step.
15. Click on Turn Cooler On in the right middle part of the UI and confirm. After few seconds, you should hear a noise caused by starting of the cooling engine.
16. Under Stirling cooler, set the temperature on channel 1 to –210 °C and wait until the actual temperature reaches it (usually around 90 min; in the meantime you can start the QCLAS instrument and let the lasers equilibrate). The power will gradually rise up to 230 W (the top limit). When the set temperature is reached, the power will decrease to approx. 110 – 120 W in order to keep the temperature.
17. Figure 4.5 shows how the UI looks when all components are connected and the Stirling cooler is switched on.

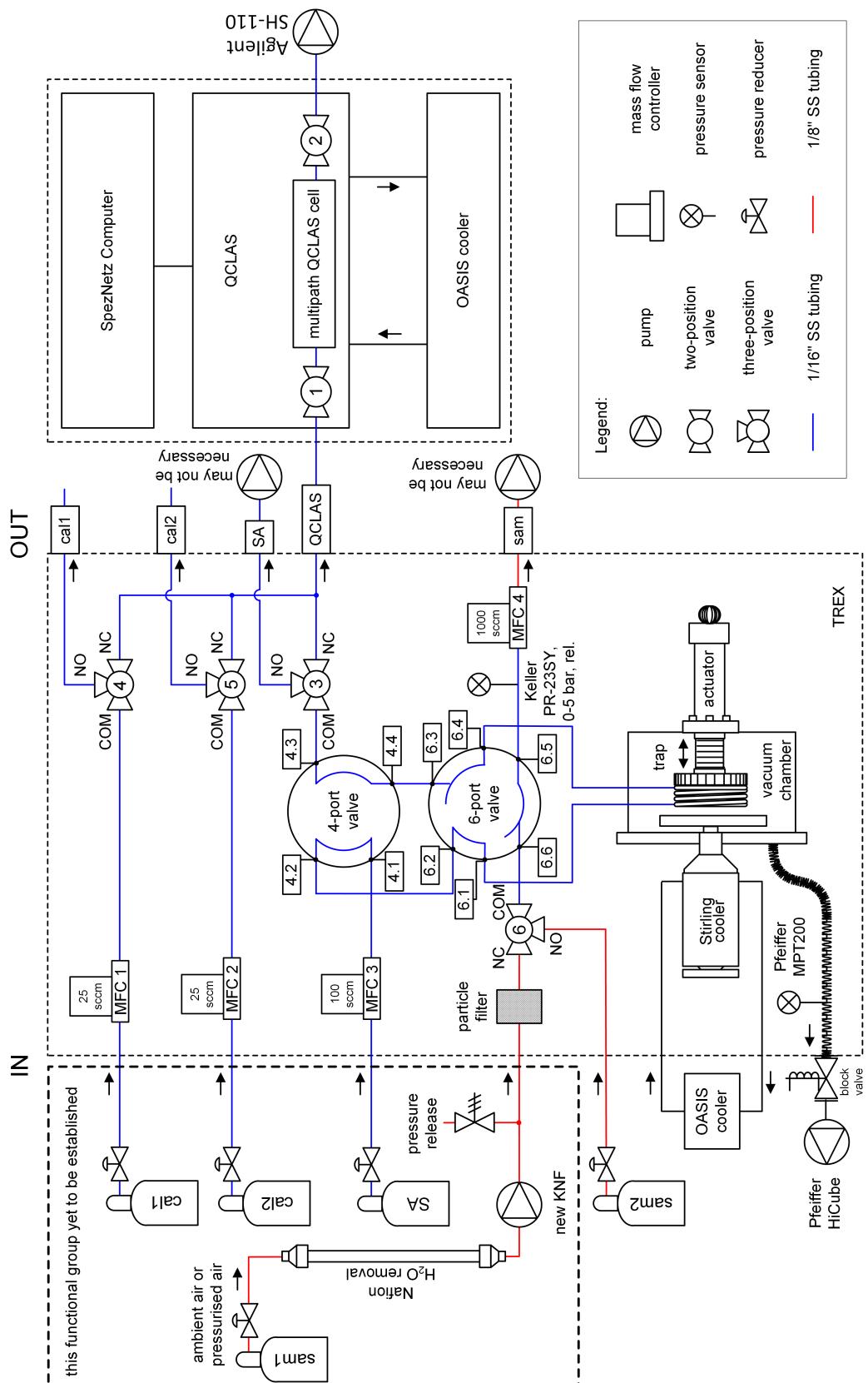


Figure 4.2: A flow scheme of the TREX-QCLAS system.

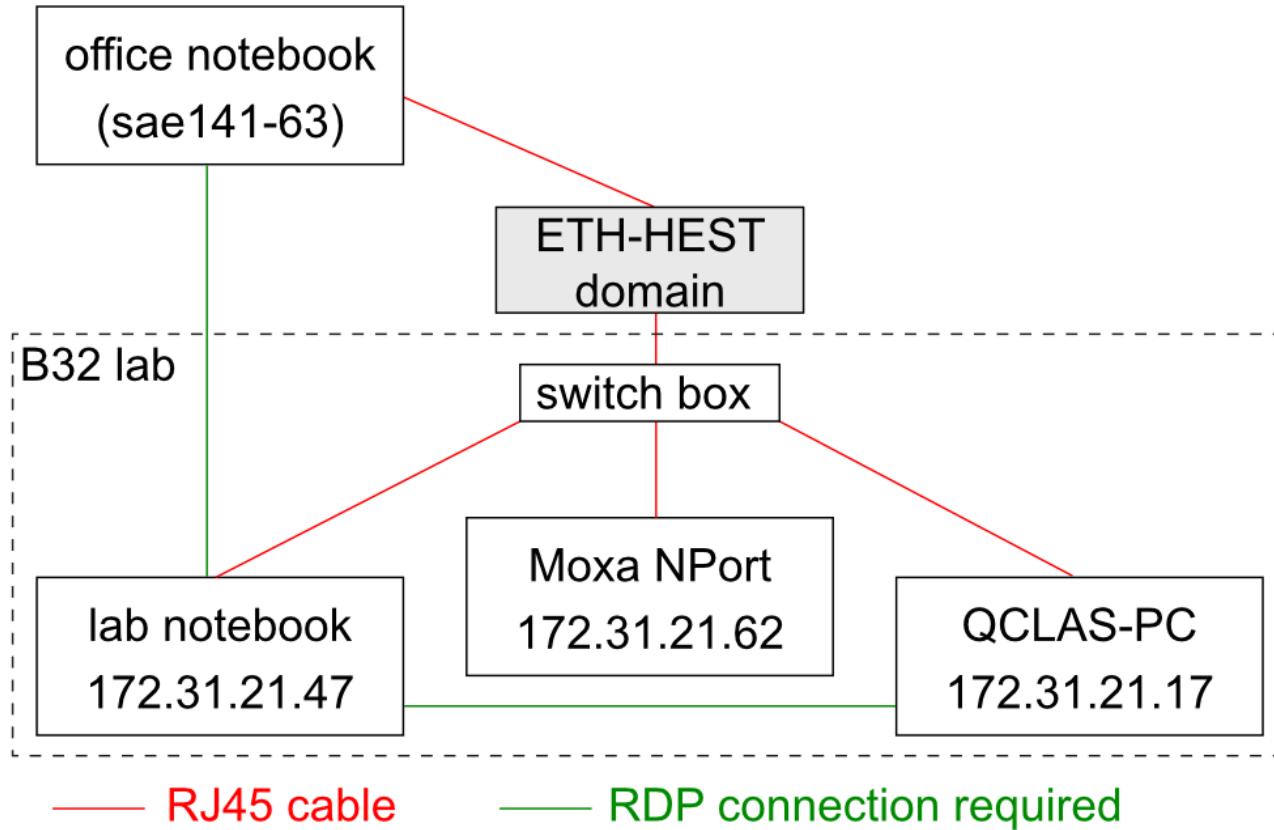


Figure 4.3: Overview of the communication setup in TREX-QCLAS. RDP stands for Remote Desktop.

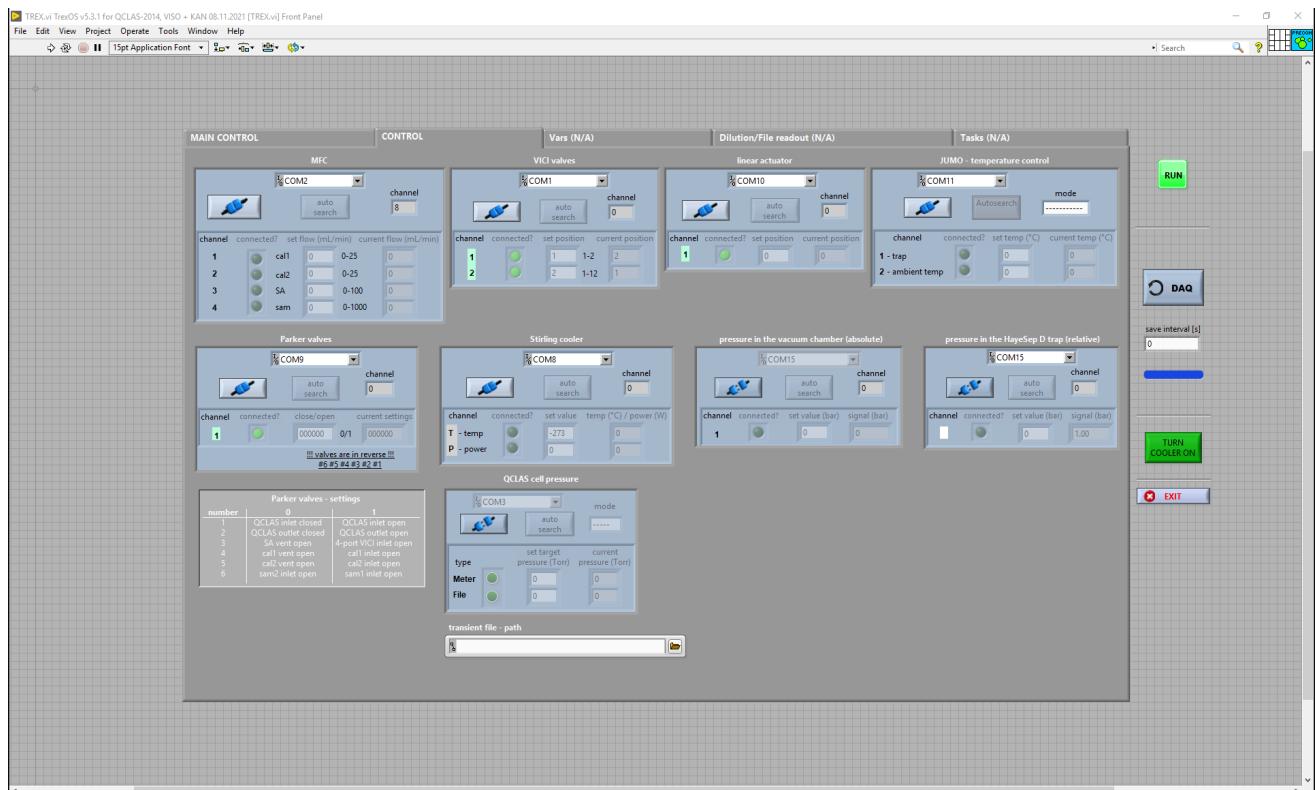


Figure 4.4: UI of TrexOS before running.

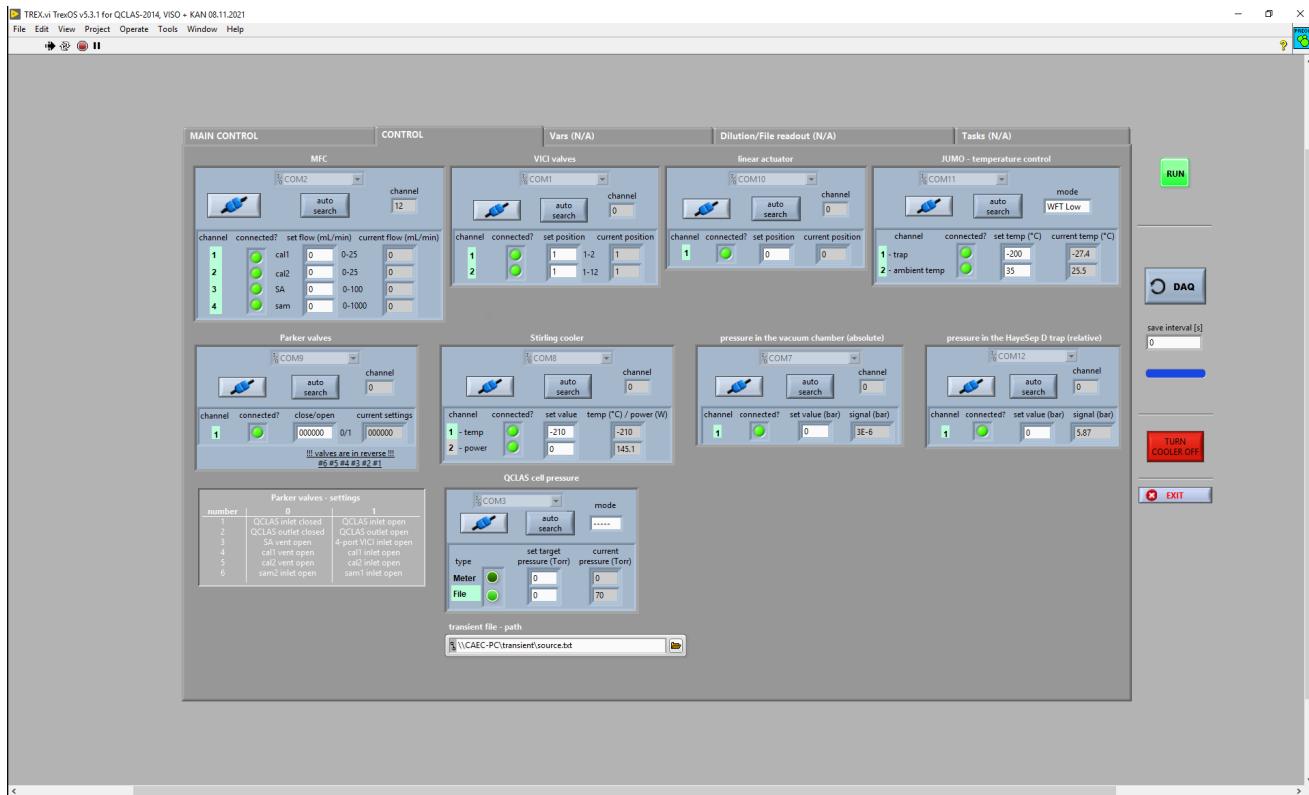


Figure 4.5: UI of TrexOS when all components are connected and the Stirling cooler is on.

4.3 Starting the QCLAS instrument

In this section, steps to start up the QCLAS instrument are described successively. Prerequisites for the start and operation of the QCLAS are:

- The QCLAS outlet is connected to the running scroll pump.
- The QCLAS cooling is connected to the external OASIS cooling unit that is set and stabilized to 14 °C. Check if the connections are not leaking.

To start the QCLAS instrument, the steps are following:

1. Turn on the switch on the back panel, next to the power cord.
2. On the front panel, check that the third diode Flow shines blue. This signalizes that the cooling provided by the OASIS unit works fine. You will not be able to turn on lasers if the diode shines red (no flow, or flow in the wrong direction).
3. Turn on the QCLAS computer by pressing the big button next to the diodes.
4. After a while, open Remote Desktop Connection on the lab notebook (on the bottom bar). Connect to the QCLAS computer – IP address: 172.31.21.17:64000, Windows username: ISG account, Windows password: abcd.1234.

5. On the QCLAS computer, start TDLWintel by opening the `TDLWintel` folder on the Desktop and the `TDLWintel14.89mx.exe` file in there. The lasers will switch on automatically, or there will be a warning message "Laser temp too high! Do you want to reactivate laser?". Choose "No" and load a recent configuration file (upper left corner). Then, let the lasers start and equilibrate for approx. 10 min.
6. On the lab notebook under File Explorer, activate the network connection between the QCLAS and lab computers by clicking on CAEC-PC under Network and fill in the log-in details from #4 if required. Without this step, the data saving in TrexOS does not work and the software produces an unclear error.

4.4 Operation of TREX-QCLAS for measurements

1. In TDLWintel in the upper left corner, click into the yellow window showing Current signal and switch to Stream data (Fig. 4.6). The blue fit line will appear on the signal screen. In the bottom left corner, activate the `f1k4` button. If the cell is filled with N₂O, check how well the blue fit line overlays the green signal line. If there are some inconsistencies, see Chapter 6.2.2.
2. If the fit is good, start saving data by activating the `wd` and `ass` buttons.
3. In TrexOS, check that the path to the transient file under `QCLAS cell pressure` is set correctly to: `\CAEC-PC\transient\source.txt`. The value for the current pressure is updated every second through the transient file only if data are being saved in TDLWintel (the `wd` button activated). If there is some problem, try deleting the copy of the transient file called `temp.txt` in the Transient folder that is actually being read by TrexOS.
4. If everything works and is ready, start saving data in TrexOS by clicking on the `DAQ` button on the right upper side of the UI and choosing a directory and a name for the `*.txt` file. Output of TREX components that are not connected at the start of data acquisition will not be saved into the DAQ file. (If pressing the DAQ button does not open a "Choose or Enter Path of File" window, it might help to go to the "Vars" card and delete the content of the "DataFile" field.)
5. On the Main Control card, open the Routine Manager (the icon with two gearwheels, Fig. 2.20).
6. Select an experiment database file (`*.xml`) in the upper left path window (Fig. 2.21). If you need to do modifications in the experiment, follow the instructions in Chapter 2.2.2. Close the window by pressing the blue Return arrow in the bottom right corner (never by the `X` in the upper right corner, otherwise the window keeps hanging somewhere and it cannot be opened again).
7. Make sure that gas cylinders that are needed for your experiment and the sampling line are connected on the back panel of TREX and their valves are open.
8. To start the experiment, go to the Main Control card. In the first box, set the number of cycles that you want to run (*N* cycles)¹ and press Start Experiment. Under, you can see the remaining time. In the bottom half on the Overview or All cards, you can observe how the experiment runs.

¹The first cycle is usually different from the rest so it is better to withdraw it from the final data set and put *N* + 1 here.

9. After the experiment finishes, deactivate the `wd` and `ass` buttons in TDLWintel and the `DAQ` button in TrexOS. The DAQ data can be found in the File Explorer under `C:\Users\localadmin\Documents\ETH TREX-QCLAS\DAQ` data files. The QCLAS data are stored on the QCLAS computer (access through Remote Desktop Connection described above) on `C:\TDLWintel\Data`.
10. In the DAQ file, the QCLAS cell pressure can be saved with some delay (few seconds). Therefore, for data analysis, data related to the QCLAS cell pressure should be read from the original stc file and not from the DAQ file.

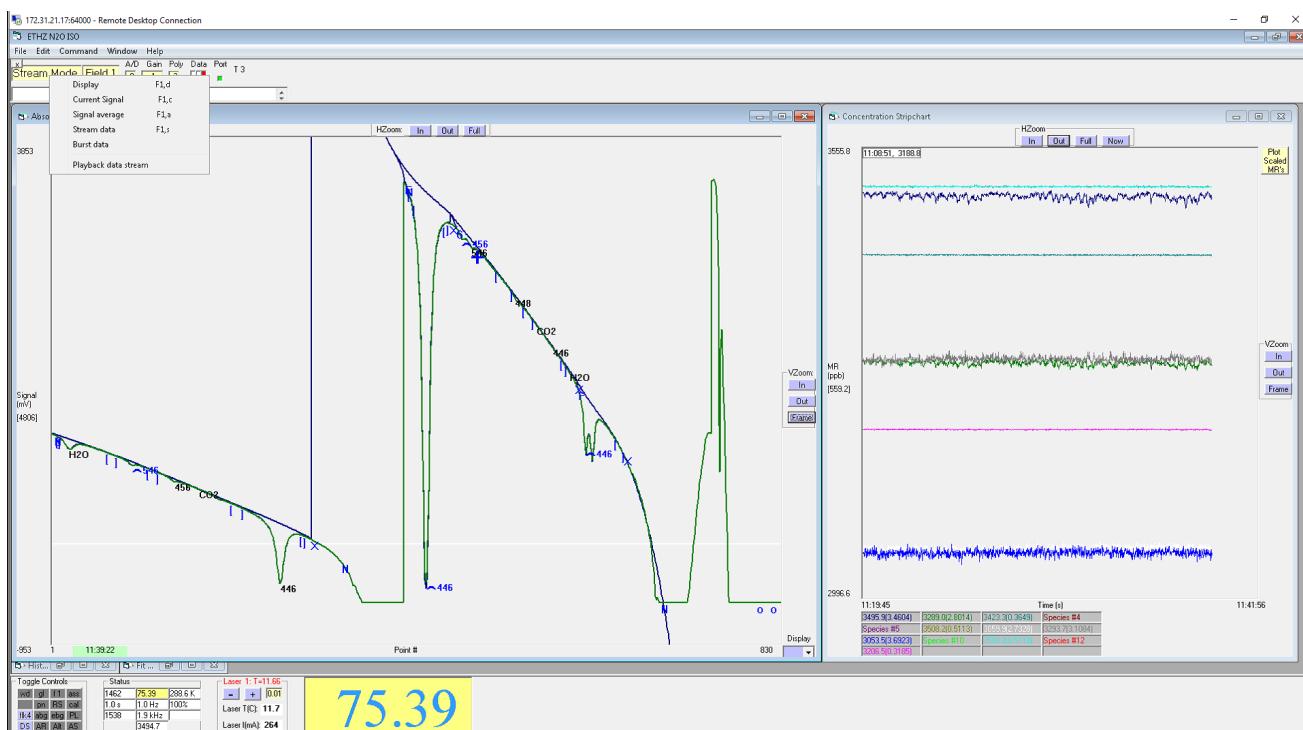


Figure 4.6: A TDLWintel printscreen showing the main window and the menu for streaming data in the upper left corner.

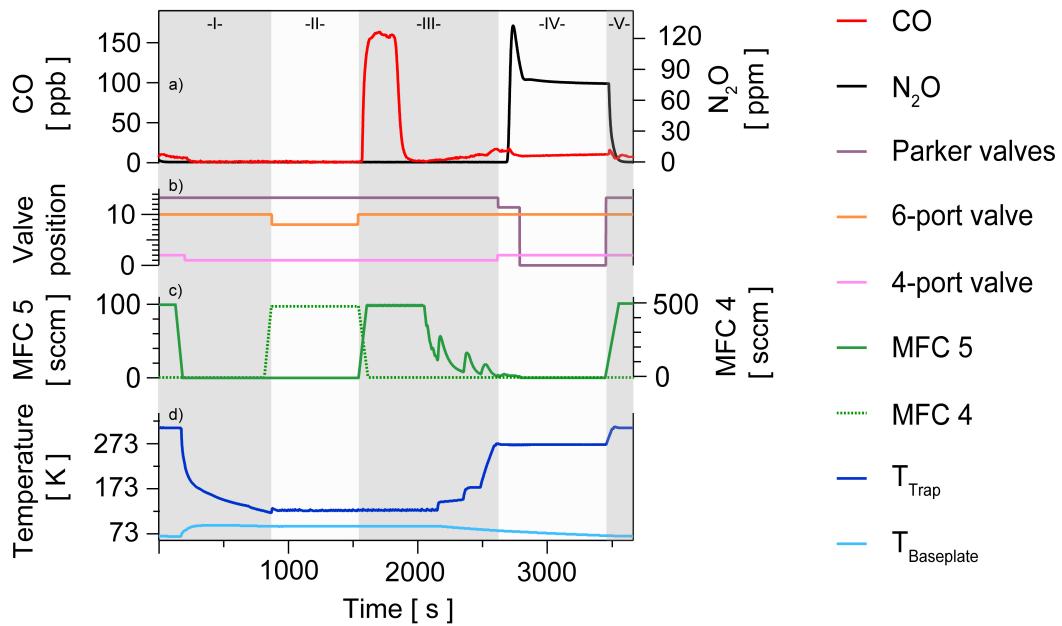


Figure 4.7: A typical course of the preconcentration routine (Ibrahim et al., 2017).

4.5 Shutting down the TREX-QCLAS system

The shut down the TREX-QCLAS system, the steps are following:

1. First, make sure that the gas cylinders are closed.
2. In TDLWintel, check that data saving is disabled. Close the software in the top right corner – the lasers will be switched off automatically.
3. Shut down the QCLAS computer.
4. Turn off the switch on the back panel of QCLAS, next to the power cord.
5. Turn off the membrane pump.
6. Turn off the OASIS cooling unit connected to the QCLAS instrument.
7. In TrexOS, check that DAQ data saving is disabled.
8. Check the temperature of the base plate of the Stirling cooler. If it is below -213°C , do not switch the Stirling cooler off, otherwise the cooler can energize itself during shutdown and become an engine (the device's manual, p. 15). Set the temperature a bit higher and wait until the base plate gets above -213°C . Only then switch the Stirling cooler off using the red button **Turn Cooler Off** in the right middle part of the UI. You should hear the Stirling engine stopping after few seconds.
9. Close TrexOS by first pressing the **Exit** button in the right middle part of the UI (important for creation of an initialization file for the next start) and then exiting LabVIEW in the top right corner.
10. Stop the turbomolecular pump by pressing its bottom right button.

11. Wait until the pump stops completely (on the display: 309 : 0 Hz, Fig. 4.8). You can speed it up by carefully aerating the pump with opening a bit the black screw on the back side of the pump – Fig. 4.9. This will fill also the vacuum chamber with air. If you want to prevent that for any reason, close the block valve by turning off the BV switch on the TREX front panel. Never let atmospheric pressure hit the running turbomolecular pump, it will be destroyed!
12. On the TREX front panel, turn off the switches Heating, Cooler, BV, and the big switch for the SimStep linear actuator.
13. Turn off the Main switch on the front panel and then the switch on the back panel, next to the power cord.
14. Finally, turn off the OASIS cooling unit connected to TREX.

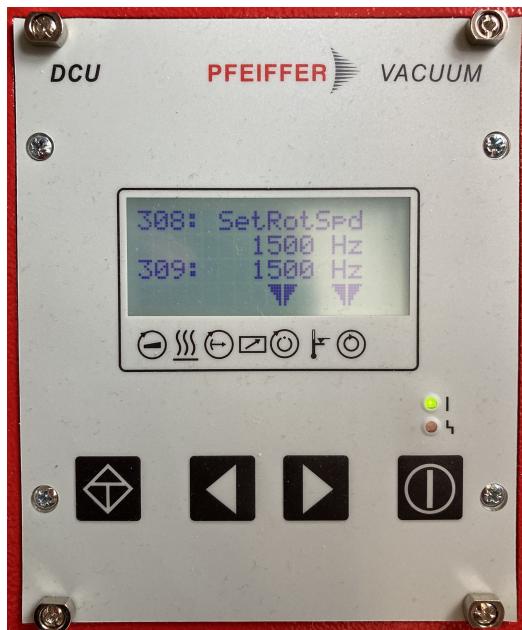


Figure 4.8: A display of the turbomolecular pump during operation.

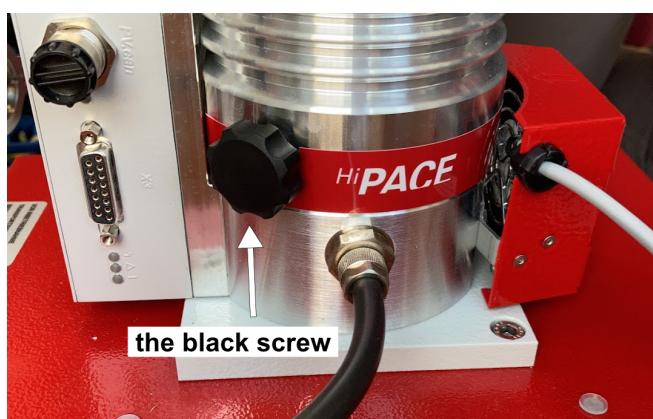


Figure 4.9: The black screw for quicker aerating the turbomolecular pump after it has been switched off.

4.6 Remote connection

If you need to connect to the instruments remotely, there are two options – through the TeamViewer software, or using Remote Desktop Connection.

- Remote connection via TeamViewer

1. connection to the lab notebook (TREX)

TeamViewer ID: 1 295 688 911

password: TREX.1234

Windows username: localadmin

password: abcd.1234

2. connection to the QCLAS computer

– there is currently no possible connection via TeamViewer to the QCLAS computer, as the Internet connection on the QCLAS computer does not work

- Remote Desktop Connection

In order to connect from outside of ETH to the instruments, first you need to be connected to the ETH network using the Cisco AnyConnect VPN client and your ETH credentials (Fig. 4.10 and 4.11). The instruments are on a more restricted hest2 network so prior to be able to connect to it, you have to ask the IT department to provide you rights to connect to the hest2 network (the common VPN network is staff-net).

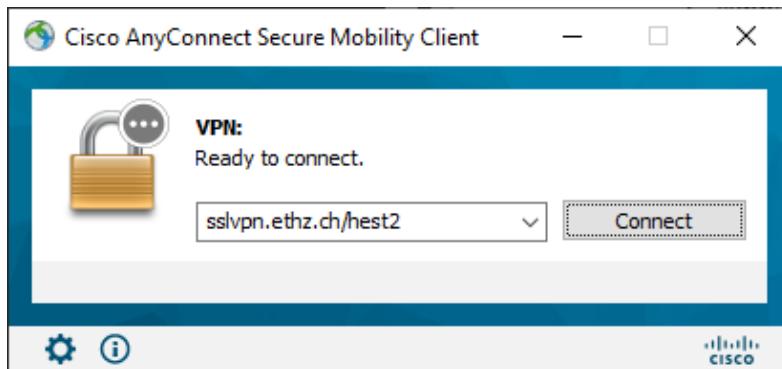


Figure 4.10: The connect screen of the Cisco AnyConnect VPN client.

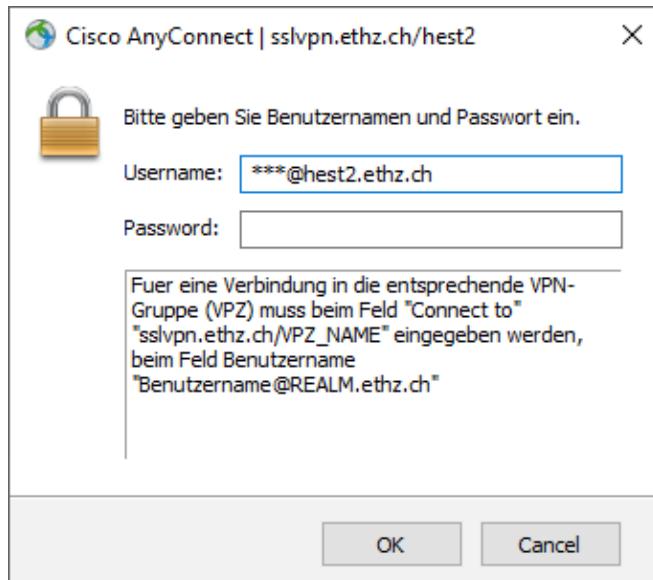


Figure 4.11: The login screen of the Cisco AnyConnect VPN client.

After you have connected to the VPN hest2 network, open Remote Desktop Connection.

1. connection to the lab notebook (TREX)

IP address: 172.31.21.47:64000

Windows username: localadmin

password: abcd.1234

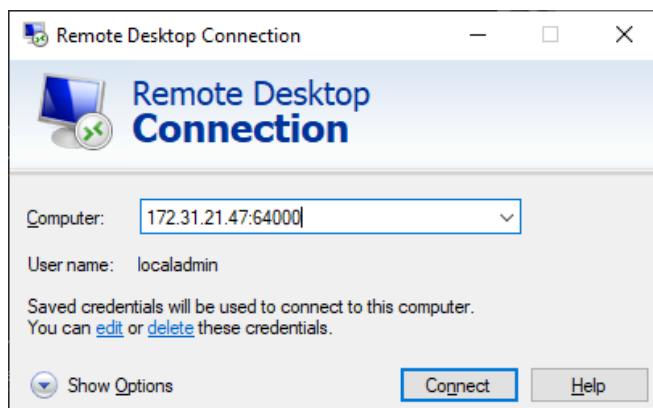


Figure 4.12: The login screen of the Remote Desktop Connection to the lab notebook.

2. connection to the QCLAS computer

In my case, the Remote Desktop Connection from my office notebook to the QCLAS computer does not work. If you wish to have a direct connection, the network settings would have to be modified by the IT department. However, you can connect first to the lab notebook as described above and then to the QCLAS computer (the Remote Desktop Connection to the QCLAS computer is usually open on the lab notebook):

IP address: 172.31.21.17:64000

Windows username: ISG account

password: abcd.1234

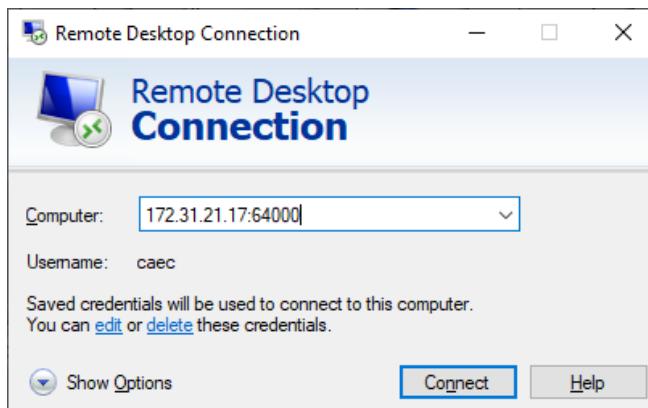


Figure 4.13: The login screen of the Remote Desktop Connection to the QCLAS computer from the lab notebook.

4.7 Time synchronization

Both computers (the QCLAS computer and the lab notebook) have for an unknown reason problems synchronizing time using Internet servers. In addition, the lab notebook loses time (every day few seconds). For analysis of the DAQ, str, and stc files, it is important to have the time stamp on both computers identical. Therefore, on the QCLAS computer, there is a batch script that is set to be executed every hour and update time on the QCLAS computer using time of the lab notebook, which is connected in the same network. The file is called "time_sync_TREX_QCLAS.bat" and stored under C:\. The content of the batch script is:

```
net time \\SAE31-21-47 /set /yes
```

The execution is set on the QCLAS computer using Windows Task Scheduler:

Create Task

- General:

Name: "Synchronize time between QCLAS and TREX"

Security options: Run whether user is logged on or not

- Triggers:

Settings: Daily, Recur every 1 day

Advanced settings: Repeat task every 1 hour for a duration of: Indefinitely

- Actions:

Action: Start a program

Program/script: C:\time_sync_TREX_QCLAS.bat

- Conditions: none

- Settings:

Allow task to be run on demand

Run task as soon as possible after a scheduled start is missed

If the running task does not end when requested, force it to stop

5 Preconcentration routine

This chapter describes an example of a preconcentration routine. The used example is based on the file `20211021_test_fractionation_HIGH_vs_precon_LOW.xml`, where the gas ETHZSAELOW was measured as a sample and the gas ETHZSAEHIGH was measured as a reference.

5.1 Routine "ResetALL"

The experiment starts with resetting all variables to starting values.

start / s	command	channel	value
0	Vici Write	1	1
0.1	Vici Write	2	1
0.2	Jumo Write	1	-200.00
0.3	VG Write	1	0
0.4	Cryotel Write	1	-210.00
0.5	MFC Write	1	1.00
0.6	MFC Write	2	0.00
0.7	MFC Write	3	1.00
0.8	MFC Write	4	1.00
0.9	Parker Write	1	35 = 100011
1	Jumo Write	2	45.00

5.2 Routine "A_S1"

In this part, a reference gas is measured meanwhile the trap is cooled down, then a sample adsorption takes place, the cell is cleaned, the adsorption stops, then the trap is purged by synthetic air (SA). Afterwards, the trap is heated to -130°C and -100°C while being purged by SA to remove co-adsorbed trace gases with lower boiling point, and finally to 0°C to desorb the preconcentrated N_2O . The gas is transferred to the optical cell and the cell is pressurized by SA to the target pressure. While the gas is being measured, the trap is heated to 35°C and cleaned by synthetic air by alternating front flush and back flush.

5.2.1 Procedure "InitSample"

start / s	command	channel	value
0	VG Write	1	6400
0.1	MFC Write	4	10.00
0.2	WaitForTemp	0	-148.00

5.2.2 Procedure "CleanCell_S"

start / s	command	channel	value
0	MFC Write	3	10.00
17	MFC Write	3	100.00
17.1	Parker Write	1	37 = 100101
27	MFC Write	3	10.00
27.1	Parker Write	1	35 = 100011
52	MFC Write	3	100.00
52.1	Parker Write	1	37 = 100101
62	MFC Write	3	10.00
62.1	Parker Write	1	35 = 100011
87	MFC Write	3	100.00
87.1	Parker Write	1	37 = 100101
97	MFC Write	3	10.00
97.1	Parker Write	1	35 = 100011
119	MFC Write	3	100.00
119.1	Parker Write	1	37 = 100101
129	MFC Write	3	10.00
129.1	Parker Write	1	35 = 100011
154	MFC Write	3	100.00
154.1	Parker Write	1	37 = 100101
164	MFC Write	3	10.00
164.1	Parker Write	1	35 = 100011
224	DoNothing	0	0

5.2.3 Procedure "Fill_cal1_S"

start / s	command	channel	value
0	MFC Write	1	3.00
0.1	Parker Write	1	43 = 101011
20	Parker Write	1	35 = 100011
105	Parker Write	1	41 = 101001
106	MFC Write	1	25.00
191	WaitCellPres	1	69.50
1000	Parker Write	1	32 = 100000

5.2.4 Procedure "Set_cal1_MFC"

start / s	command	channel	value
0	MFC Write	1	1.00

5.2.5 Procedure "Set_cal1_marker"

start / s	command	channel	value
0	Set Marker	0	11
5	Set Marker	0	0

5.2.6 Procedure "Measure4Min"

start / s	command	channel	value
240	DoNothing	0	0

5.2.7 Procedure "BeginTrapSample"

start / s	command	channel	value
0	MFC Write	4	550.00
5	Vici Write	2	3

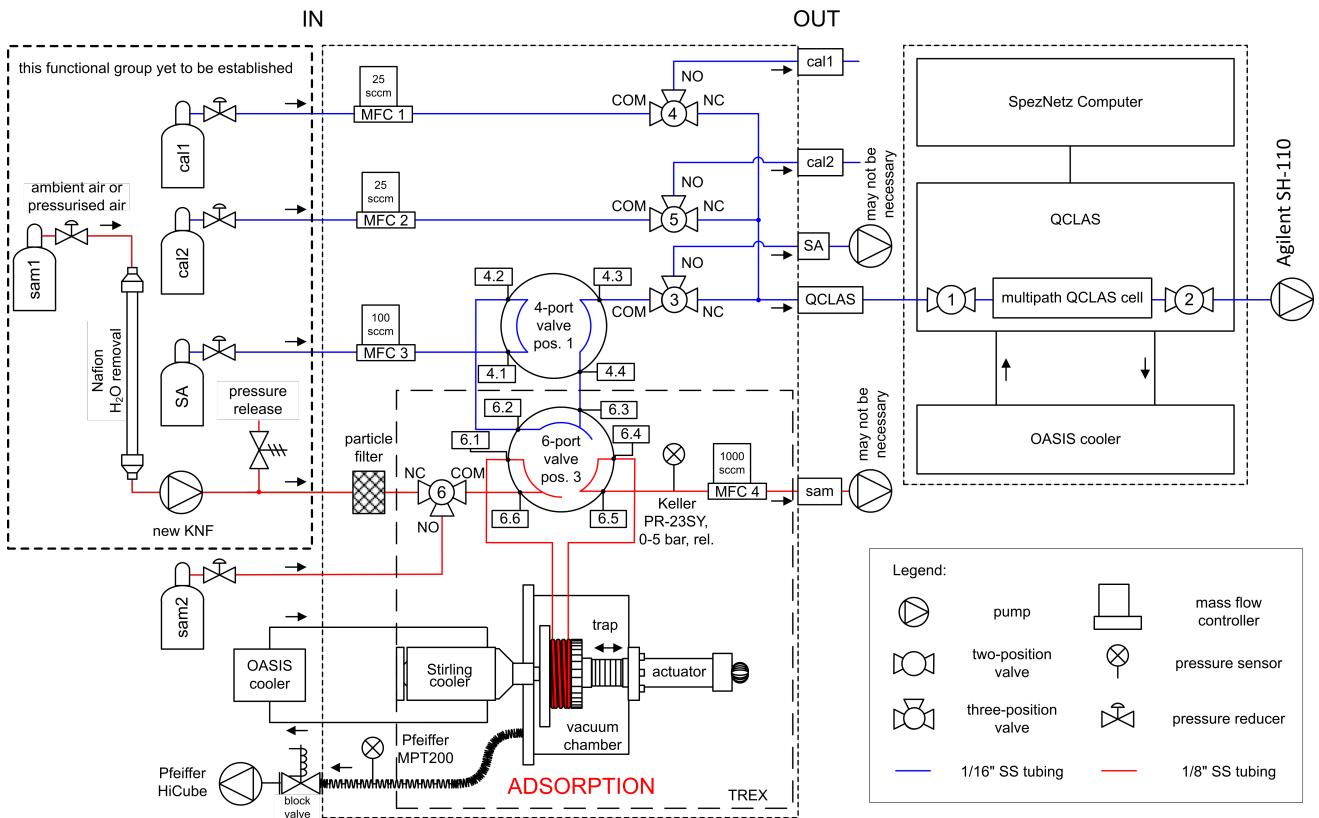


Figure 5.1: A flow scheme for the adsorption part of the N_2O preconcentration.

5.2.8 Procedure "CleanCell_S"

start / s	command	channel	value
0	MFC Write	3	10.00
17	MFC Write	3	100.00
17.1	Parker Write	1	37 = 100101
27	MFC Write	3	10.00
27.1	Parker Write	1	35 = 100011
52	MFC Write	3	100.00
52.1	Parker Write	1	37 = 100101
62	MFC Write	3	10.00
62.1	Parker Write	1	35 = 100011
87	MFC Write	3	100.00
87.1	Parker Write	1	37 = 100101
97	MFC Write	3	10.00
97.1	Parker Write	1	35 = 100011
119	MFC Write	3	100.00
119.1	Parker Write	1	37 = 100101
129	MFC Write	3	10.00
129.1	Parker Write	1	35 = 100011
154	MFC Write	3	100.00
154.1	Parker Write	1	37 = 100101
164	MFC Write	3	10.00
164.1	Parker Write	1	35 = 100011
224	DoNothing	0	0

5.2.9 Procedure "EndTrapSample"

start / s	command	channel	value
80	Vici Write	2	1
80.1	MFC Write	4	0.00
80.2	Parker Write	1	3 = 000011

5.2.10 Procedure "PurgeCO"

start / s	command	channel	value
0	VG Write	1	5000
0.1	MFC Write	3	100.00

5.2.11 Procedure "Measure3Min" (actually just purging)

start / s	command	channel	value
180	DoNothing	0	0

5.2.12 Procedure "PurgeTo130_S"

start / s	command	channel	value
0	VG Write	1	5000
0.1	MFC Write	3	100.00
0.2	Parker Write	1	3 = 000011
17	Parker Write	1	5 = 000101
30	Parker Write	1	3 = 000011
52	Parker Write	1	5 = 000101
75	Parker Write	1	3 = 000011
87	Parker Write	1	5 = 000101
97	Parker Write	1	3 = 000011
110	Parker Write	1	5 = 000101
140	Parker Write	1	3 = 000011
160	Parker Write	1	5 = 000101
180	Parker Write	1	3 = 000011
240	MFC Write	3	5.00
340	MFC Write	3	1.00

5.2.13 Procedure "HeatTo130"

start / s	command	channel	value
0	VG Write	1	0
0.1	Jumo Write	1	-130.00
0.2	MFC Write	3	1.00
180	DoNothing	0	0

5.2.14 Procedure "HeatTo100"

start / s	command	channel	value
0	VG Write	1	0
0.1	Jumo Write	1	-100.00
0.2	MFC Write	3	1.00
180	DoNothing	0	0

5.2.15 Procedure "HeatTo0"

start / s	command	channel	value
0	VG Write	1	0
0.1	Jumo Write	1	3.00
0.2	MFC Write	3	1.00
180	DoNothing	0	0

5.2.16 Procedure "DesorbSam"

start / s	command	channel	value
0	MFC Write	3	3.00
0.1	Vici Write	1	2
0.2	Vici Write	2	1
0.3	Parker Write	1	5 = 000101
15	MFC Write	3	25.00
100	WaitCellPres	1	69.50
360	Parker Write	1	0 = 000000

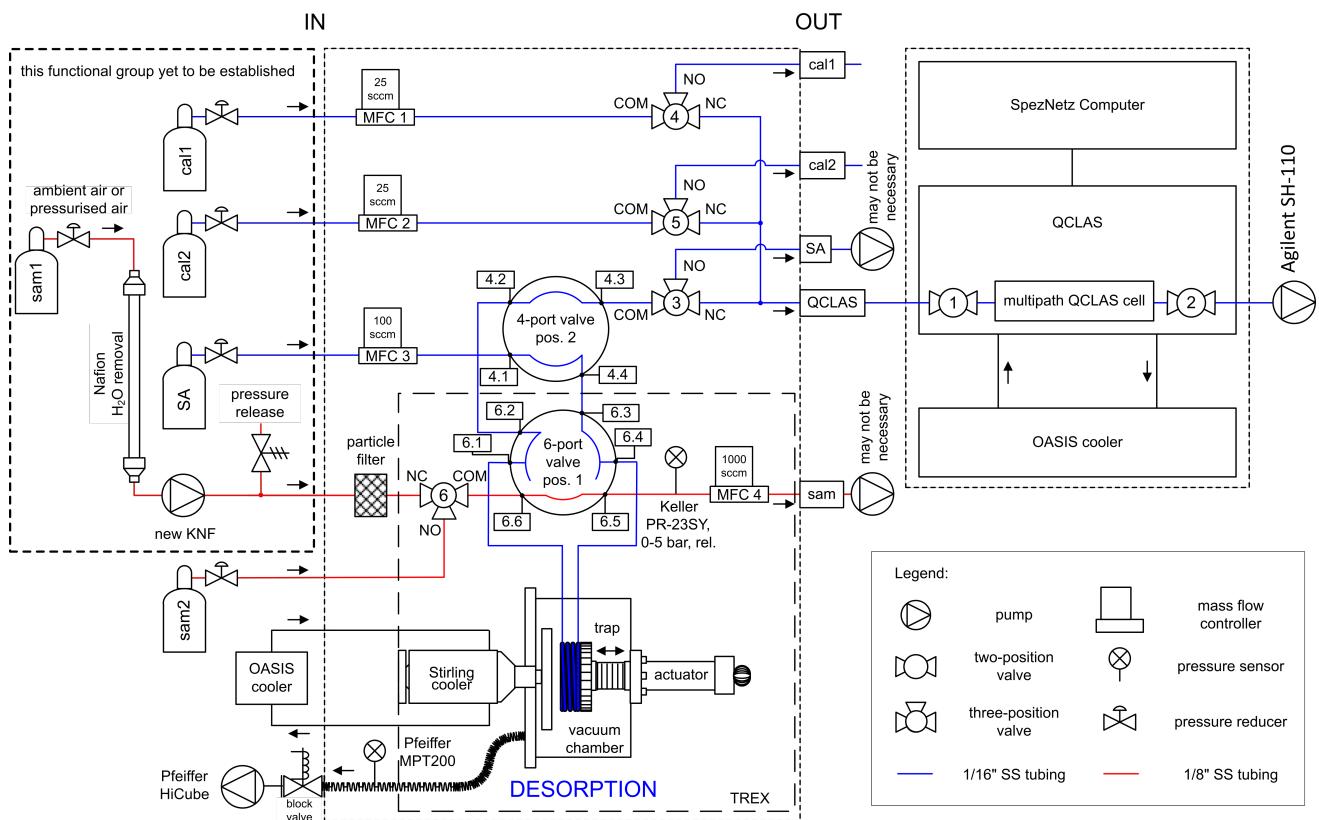


Figure 5.2: A flow scheme for the desorption part of the N_2O preconcentration.

5.2.17 Procedure "MeasureSam"

start / s	command	channel	value
0	Set Marker	0	1
5	Set Marker	0	0

5.2.18 Procedure "HeatOutSam"

start / s	command	channel	value
0	Vici Write	1	2
0.1	MFC Write	3	100.00
0.2	Jumo Write	1	35.00
100	Vici Write	1	2
200	Vici Write	1	1
300	Vici Write	1	2
400	Vici Write	1	1
500	Vici Write	1	2
600	Vici Write	1	1
660	Jumo Write	1	-200.00
660.1	MFC Write	3	1.00
660.2	Parker Write	1	0 = 000000

5.3 Routine "ResetALL_end"

In the final part, all variables are set back to default.

start / s	command	channel	value
0	Vici Write	1	1
0.1	Vici Write	2	1
0.2	Jumo Write	1	-200.00
0.3	VG Write	1	0
0.4	Cryotel Write	1	-210.00
0.5	MFC Write	1	0.00
0.6	MFC Write	2	0.00
0.7	MFC Write	3	0.00
0.8	MFC Write	4	0.00
0.9	Parker Write	1	0 = 000000
1	Jumo Write	2	45.00

6 Maintenance and troubleshooting

6.1 TREX

6.1.1 JUMO 1

During the experiment in the `InitSample` phase, it can occasionally happen that JUMO 1 will not be set to $-200\text{ }^\circ\text{C}$ (heating switched off). Even though in TrexOS it shows that it is set correctly, on the TREX front panel, one can easily check that the set point is wrong – it stays at the previous value, usually $25\text{ }^\circ\text{C}$. It manifests during the cooling of the trap while it is pressed against the base plate – first, the trap cools down as usual but then the heating switches on periodically as it tries to get it back to the previously set value. As a result, the trap is heated and temperature of the base plate goes up, too. If this happens, it has to be fixed as quickly as possible, otherwise there is a big heat transfer towards the Stirling that could damage it. It has to be fixed manually in TrexOS on the `Control` panel – set JUMO 1 to another value (e.g. $-100\text{ }^\circ\text{C}$) and then again to $-200\text{ }^\circ\text{C}$. Only then, JUMO 1 will be set correctly and the heating will switch off. Consequently, it might happen that the trap will not cool down sufficiently enough for the adsorption phase, so data from this cycle will be unusable.

*** this was most probably occurring due to too many commands being performed at the end of the experiment file: JUMO 1 being set to $-210\text{ }^\circ\text{C}$, $25\text{ }^\circ\text{C}$, and $-200\text{ }^\circ\text{C}$ within 0.5 s; this was corrected in the new experiment file on 21/10/2021 that was used in Chapter 5 -> let's see if it helps, so far it looks that this problem is solved ***

6.1.2 The trap

After longer period of non-usage, it can happen that the trap will be blocked. It will manifest as almost no flow through the trap – when VICI 1 is set to position 1 and VICI 2 to position 3 (adsorption), SA flow will be alright but sample flow will be zero. When VICI 1 is set to position 1 and VICI 2 to position 1, SA flow will work only sometimes and only a bit (around 2 mL/min or less). When VICI 1 is set to position 2 and VICI 2 to position 1 (desorption + backflush), there will be no flow of SA at all.

The solution that helped in the past is to bake the trap at $35\text{ }^\circ\text{C}$ (JUMO 1) while having set low flow of SA (MFC 3 – 10 mL/min) and evacuating through the QCLAS cell (Parker – 000111, VICI 1 – 1, VICI 2 – 1). The actual SA flow will be lower as the trap is blocked. Heating to $35\text{ }^\circ\text{C}$ and the SA flow should release the blockage after some minutes. Once the flow of SA restores to the set value and can be increased up to 100 mL/min, continue with this:

- for 5 min: VICI 1 – position 1, VICI 2 – position 1, JUMO 1 – $35\text{ }^\circ\text{C}$, MFC 3 (SA) – 100 mL/min, Parker – 000111
- for another 5 min: VICI 1 – position 2, VICI 2 – position 1, JUMO 1 – $35\text{ }^\circ\text{C}$, MFC 3 (SA) – 100 mL/min, Parker – 000111

6.1.3 The VICI valves

In the DAQ files, the VICI 1 and VICI 2 values can sometimes jump for a second from the set value to zero while the position of the valves is switched; e.g. $1 \rightarrow 0 \rightarrow 2$. This then can make a problem in the data analysis when a change in the VICI position is used as a marker. If this happens, manually edit the DAQ file and replace the zeros.

6.1.4 Opening the vacuum chamber

In case the vacuum chamber needs to be opened, first wait before the temperature of the base plate gets to RT. Otherwise water vapour from ambient air can freeze out or condense on the base-plate surface. For the actual opening of the chamber, the cables from the Moxa board need to be disconnected and the board needs to be shifted to a specific position such that all screws on the chamber can be reached and loosened.

6.2 QCLAS

When the cell is being filled by the Wait For Pressure command and the pressure is still rather low (< 10 Torr), the fit often gets off and TDLWintel would show "Reference lock fit failed to converge! Ignore results". This error goes usually away by itself when the pressure increases above 15 Torr, therefore it is suggested to first wait before interfering.

6.2.1 QCLAS – optical alignment

In case the instrument has been moved and/or the light level in TDLWintel has decreased (suddenly or gradually), it might mean that there is a problem with the optical alignment.

1. Evacuate the optical cell and keep the pressure low.
2. Open the spectrometer.
3. In TDLWintel, switch to the Current Signal mode in the upper left corner.
4. Switch the signal frequency to 10Hz by going to Commands/Quick Keystrokes/Current signal at 10 Hz or pressing Ctrl+Q.
5. On the bottom left panel in the Status windows, select the first values showing signal range,
6. Switch the signal output in the Absorption Spectrum window from detector 1 to detector 2 by clicking onto the A/D window in the upper left corner ($0 \rightarrow 1$). This shows the light level without going through the cell (the reference path in Fig. 3.2).
7. Manually flip in the pinhole (between the IR-IR combining mirror and the beamsplitter; Fig. 3.2).
8. Adjust the horizontal and vertical zooms of the shown spectrum if needed, by clicking on the Horizontal Full button and then the Vertical Frame button.
9. With a hex key size 2 mm, adjust the focus of both laser objectives onto the pinhole. Slightly turn the X-Y-Z screws on both objective stages (repeating it few times in the same order) until the light level output is maximized for both lasers (the signal range in the big yellow window in the bottom of TDLWintel).

10. Flip out the pinhole.
11. In TDLWintel, switch the signal output from detector 2 back to detector 1 ($1 \rightarrow 0$). This shows again the light going through the cell.
12. Adjust the horizontal and vertical zooms of the shown spectrum if needed.
13. With a hex key size 2.5 mm, slightly adjust mirrors in a certain sequence few times to maximize the light level. The sequence is M3 – M9 – M4 – M9 – M3 – M9 – etc. Adjust always both screws on a mirror before moving to the next one.
14. When the light levels are maximized, switch the signal frequency back to 1 Hz by going to Commands/Quick Keystrokes/Refit time to 1 sec or pressing Ctrl+O.
15. Save the new laser parameters by going to Command/Kopy konditions to klipboard or pressing Ctrl+K. Paste it to the Note.txt file under C:\TDLWintel\Notes and Docs and save it.
16. The spectrometer can be closed again.

6.2.2 QCLAS – improve fit

If the fit goes bad, the tuning rate of the lasers needs to be either only slightly modified, or renewed completely. For a slight modification of the tuning rate, see the last three points of the procedure below. In case the tuning rate needs to be updated completely, follow the full procedure described below. If something is unclear, you can consult also the TDLWintel manual.

1. In TDLWintel, switch to the Current Signal mode in the upper left corner.
2. In the bottom left corner, undo the flk4 button if it is on.
3. Switch the signal output in the Absorption Spectrum window from detector 1 to detector 2 by clicking onto the A/D window in the upper left corner ($0 \rightarrow 1$). This shows the light level without going through the cell (the reference path in Fig. 3.2).
4. Flip in the etalon and the flag by going to Commands/Operate Valves and Switches and click Open under ETALON and FLAG.
5. Adjust the horizontal and vertical zooms of the shown spectrum if needed. Figure 6.1 shows how the output spectrum should look like.
6. Transfer the spectrum parameters from Field 1 to Field 2 by going to Command/Transfer field parameters and selecting All of the above, Field #1, and Field #2 and pressing Implement.
7. Remove the etalon by going to Commands/Operate Valves and Switches and click Close under ETALON.
8. Adjust the horizontal and vertical zooms of the shown spectrum if needed. Figure 6.2 shows how the output spectrum should look like now.
9. Switch to the Display mode in the upper left corner.

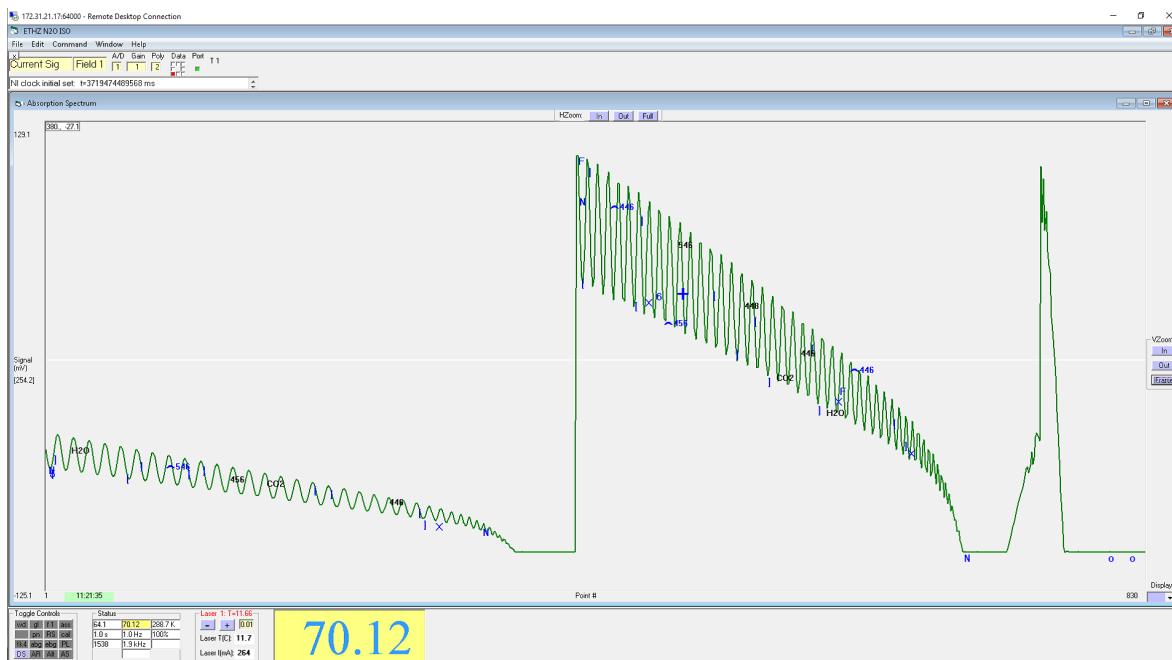


Figure 6.1: A typical etalon spectrum.

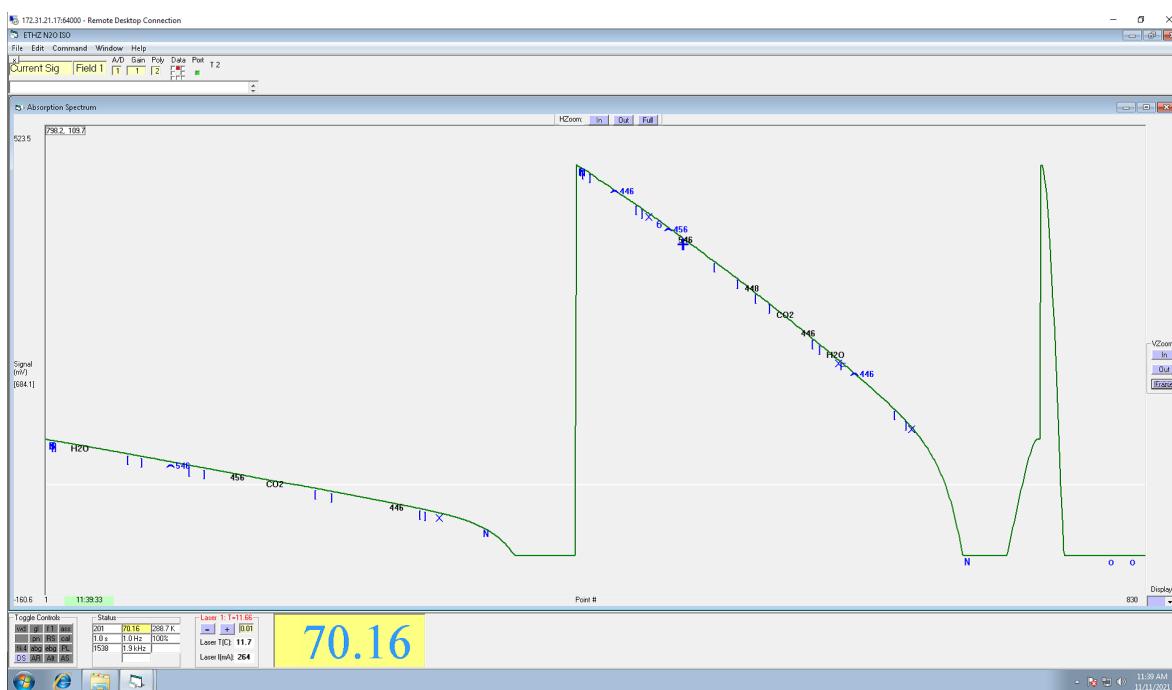


Figure 6.2: A typical spectrum after taking the ratio of Field 1 and Field 2.

10. Remove the flag by going to Commands/Operate Valves and Switches and click Close under FLAG.
11. Take ratio between spectra in Field 1 and Field 2 by going to Command/Ratio, subtract, add, filling in Field 1 = Field 1 / Field 2 and pressing Implement. The spectrum will now look similar to Fig. 6.1.

12. Fit the etalon spectrum by going to Command/Get tune rate from etalon fit.
13. Check that there is always one white \wedge marker in the position of each minimum and maximum of the spectrum (in between the N symbols representing the start and the end of the etalon fit) and that the yellow vertical lines go through each minimum and maximum (positions of the markers and the lines may not be exactly identical).
14. If the fit is good, continue to the next point. If not, consult the TDLWintel manual (p. 32 and p. 76, Appendix J). In Field 1, there is already the new tuning rate applied.
15. Transfer the new tuning rate also to other fields by going to Commands/Duplicate tuning rates or pressing Ctrl+U.
16. Return to the Current Signal mode in the upper left corner.
17. Switch back the signal output from detector 2 to detector 1 ($1 \rightarrow 0$). This shows again the light going through the cell.
18. If the cell is filled, switch to the Stream Mode, turn on the flk4 button.
19. Evaluate the fit to see if the peaks in the fit agree well with the measured peaks. If not, this can be improved by slightly stretching or shrinking the tuning rate. This can be done by going to Edit/Tuning Rate Params, right-clicking the + or - button in the Stretch/Shrink Tune Rate field, and observing the spectrum/fit response. You can also monitor the χ^2 values of individual fits under Window/Fit Report (the lower, the better).
20. When the optimization is finished, transfer again the modified tuning rate to other fields by going to Commands/Duplicate tuning rates or pressing Ctrl+U.
21. Save the new instrument configuration under File/Save configuration as.

6.3 TrexOS

In case the lab notebook or the QCLAS computer were switched off prior to a measurement, there will be no connection between the two computers in File Explorer and the loading of the transient file in TrexOS will not work. On the lab notebook, open the CAEC-PC under Network and log in. Now the connection is active.

After longer period of usage, it can happen that the reading of the transient file in TrexOS becomes too slow – the values on the Main Control card are updated only every few seconds instead of every second and the Wait For Pressure command during the cell filling often overshoots the set value due to the slow reading of the actual value. If this occurs, it means that the transient file got too big (close to 100 MB or more). Stop the data writing in TDLWintel if it is on (the wd button). Go to the CAEC-PC/transient folder and delete both source.txt and temp.txt files. Start the data writing in TDLWintel again. In TrexOS on the Control card, load the newly created transient file by pressing the auto search button under QCLAS cell pressure. The reading of the file will start and it should be again fast.

6.4 The whole system

The cooling liquid for the OASIS coolers consists of 80 vol. % H₂O and 20 vol. % ethanol. There is a bottle with already prepared cooling liquid in B32 in one of the bottom cabinets. The level of cooling liquid in all coolers should be checked regularly and adjusted if needed.

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List of abbreviations

- IR – infrared
- PSU – power supply unit
- QCL – quantum cascade laser
- QCLAS – quantum cascade laser absorption spectroscopy
- RDP – Remote Desktop connection
- RT – room temperature
- SA – synthetic air
- TDL – tunable diode laser
- TREX – TRace gas EXtractor
- TrexOS – TREX operation software