How to use the Autoclave

This is an introductory guide on how to use the autoclave and external gas cylinders that are placed in HG03-100 GC 2 (the gas cylinder cabinet outside of lab 03.118). In this guide, we will refer to opening and closing valves, and with this it is meant that the valve is completely open/closed (turn it until it does not go any further, do not stop half way!!!).

It is very important when using this equipment that you remember the rule “righty thighty, lefty loosey” for almost all valves (exceptions will be stated)

Necessary equipment:

To use the Autoclave, you need the following things:

- The autoclave (obviously)
- Tongs/tweezers
- Spanners (size 41 and red marked spanner from lab 03.115)
- Teflon tape
- Vice in lab 03.113
- Labjack
- Working Schlenkline

Make sure that you have all materials at hand and that you have asked the person working in fumehood 03.115 H9, if you can use half of his working space, because the gas pipes only lead to this fumehood and therefore it is the only place you can connect the autoclave and fill it.

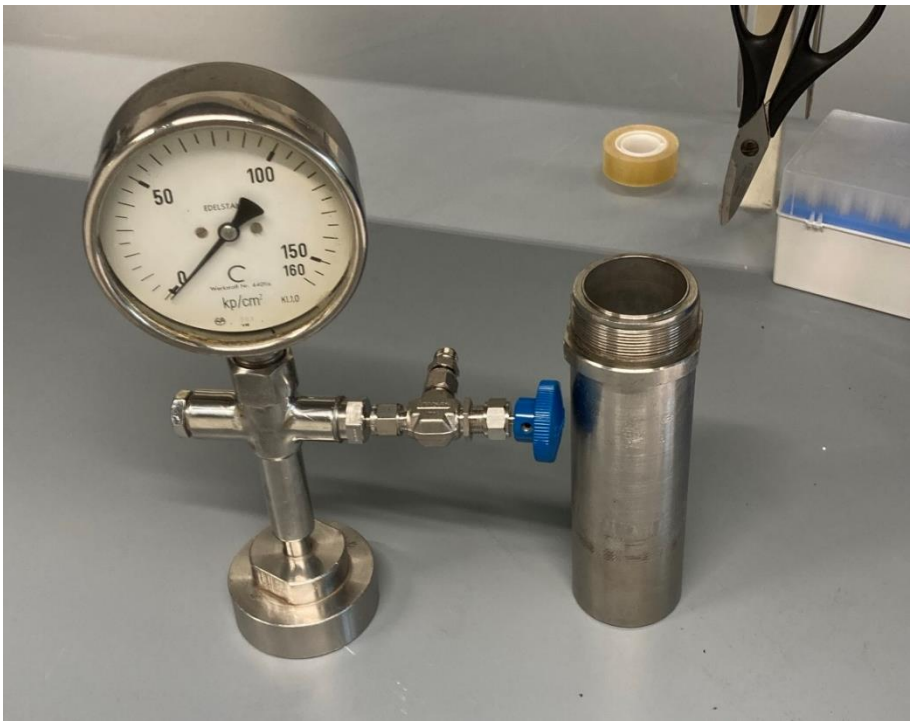

Figure 1: Autoclave top part (left) and bottom part (right)

First, you need to prepare the bottom cylinder of the autoclave. Wrap the top part of the cylinder tight with Teflon tape **once**. Make sure that you wrap the Teflon tape as close to the top edge as possible but not over it (see figure 3). The reason for using the Teflon is that the seal of the barometer-part of the autoclave is not perfect anymore. (It is not broken, but you will lose a significant part of your pressure over time, especially overnight). It is also very important that you only wrap it once, because otherwise you will not be able to close the autoclave anymore.

Figure 2: Teflon tape

Figure 3: Teflon tape wrapped just to the edge of the opening

Now, prepare your reaction mixture in a glass vial and put it into the cylinder of the autoclave (best using tongs or tweezers). The maximal reaction volume is 2 mL. It is also possible to directly fill the autoclave, but found it more difficult to clean up and retrieve my product.

Close the Autoclave using your hands and take it to Lab 03.113 (Note: **most of the autoclave's weight is now resting at the top and it can quickly fall over. Pay very close attention to that, because the barometer may break, if it falls.**)

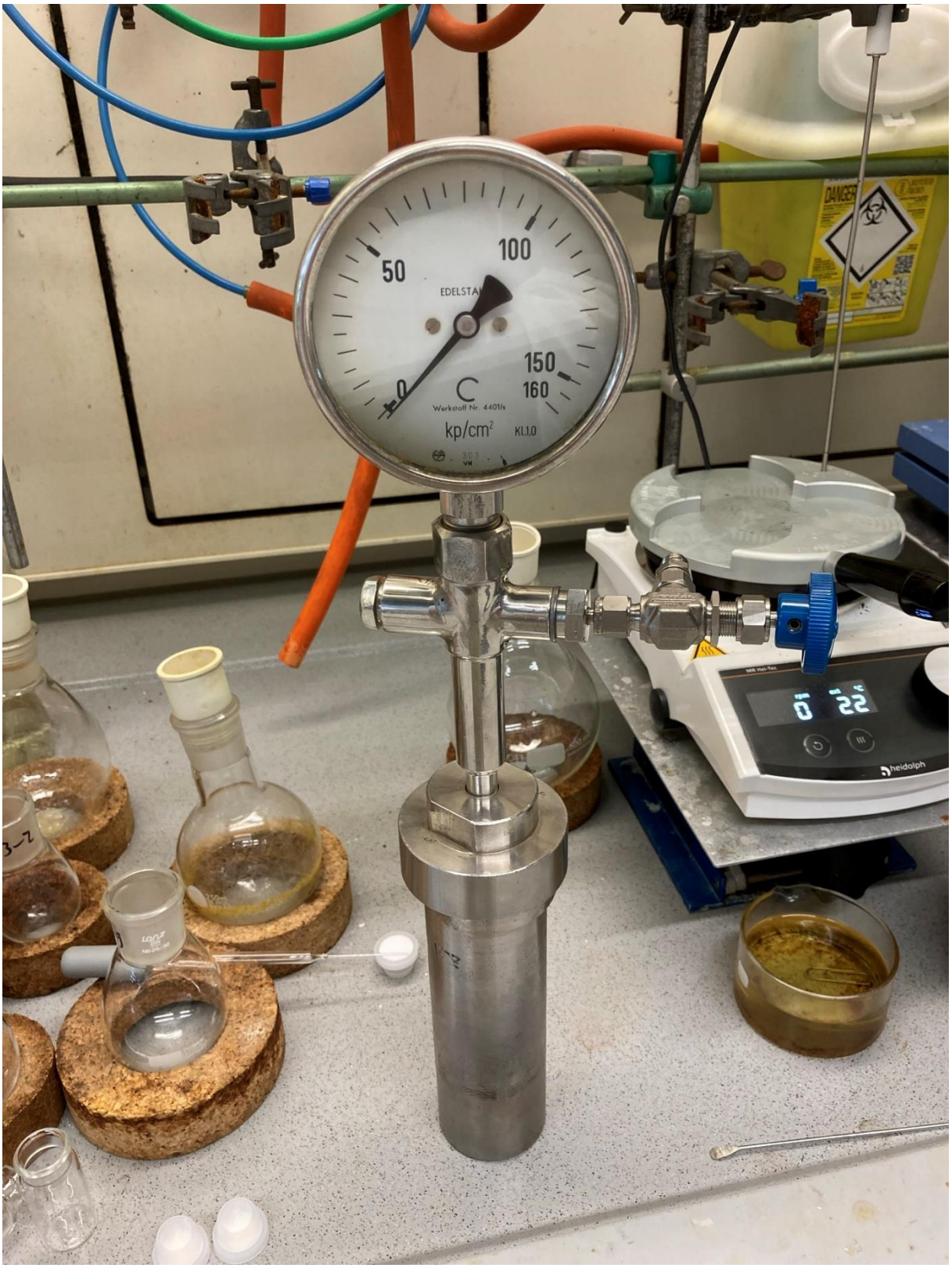

Figure 4: Closed autoclave. Pay attention because most of the weight is now resting at the top and it can quickly fall over.

There, you can find a vice and a spanner size 41. Put the Autoclave into the vice. Make sure that you use the flat sites on the cylinder part of the autoclave and make sure that you secure it very tightly using the weight of your body, if necessary.

Figure 5: Equipment found in lab 03.113

Figure 6: The autoclave is secured on the flat edges of the bottom part in the vice

Close the autoclave using the spanner as tight as you can (using the weight of your body if necessary).

Figure 7: Closing the autoclave using the Spanner size 41

Then, go back to your fumehood and connect the autoclave to a clamp so that it does not fall over and then to your schlenkline. Make sure that the vacuum is connected to the house-vacuum (and NOT your oil pump, because that one is too strong and you would evaporate your solvent/splash everything around in the cylinder).

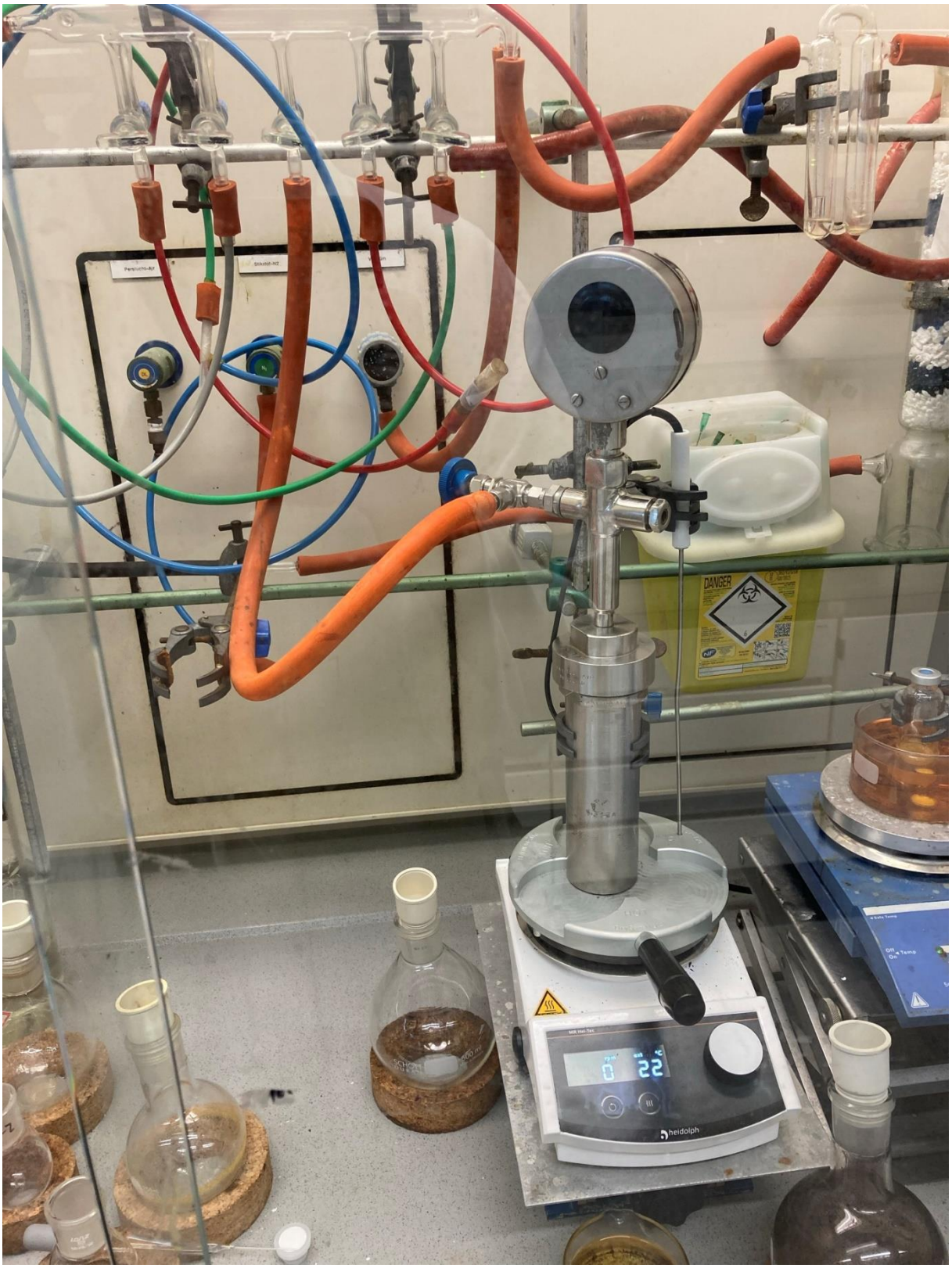

Figure 8: Autoclave connected to the Schlenkline

Open the blue valve on the autoclave and quickly evacuate and backfill with argon 3x. (It is recommended to turn the stopcock of the schlenkline at a moderate speed. We do not want full vacuum, just a replacement of most of the air with argon, by sucking out a little bit and backfilling it with argon). Then close the blue valve again. (Note: Considering your catalyst and danger of said

catalyst (Raney Nickel is a pyrophoric solid for example), you may want to evacuate first, before you go to the vice and tightly close the autoclave)

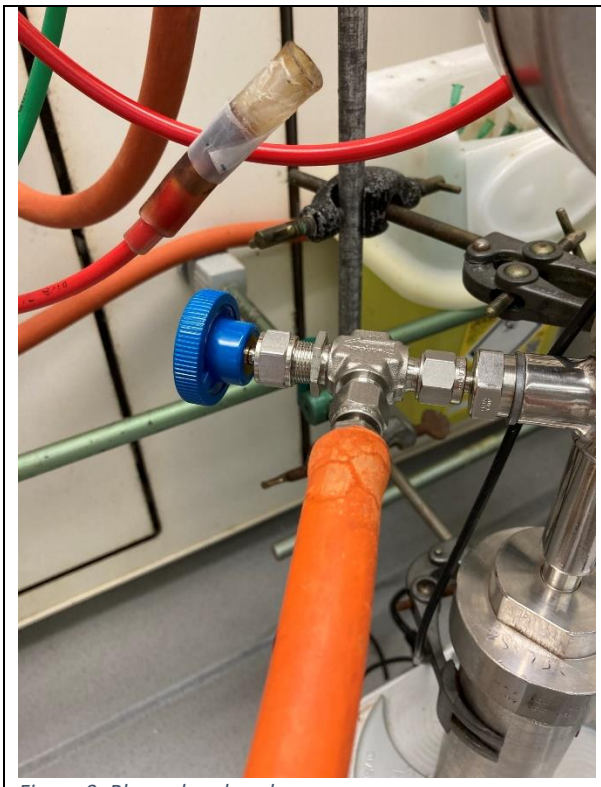

Figure 9: Blue valve closed

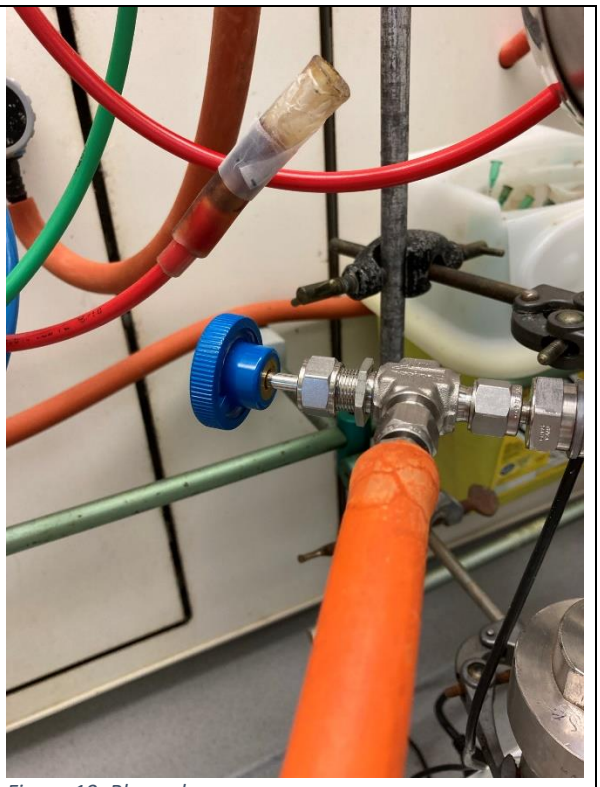

Figure 10: Blue valve open

Now go to fumehood 03.115 H9 and connect the autoclave to the pressure outlet. Elevate a labjack to support it.

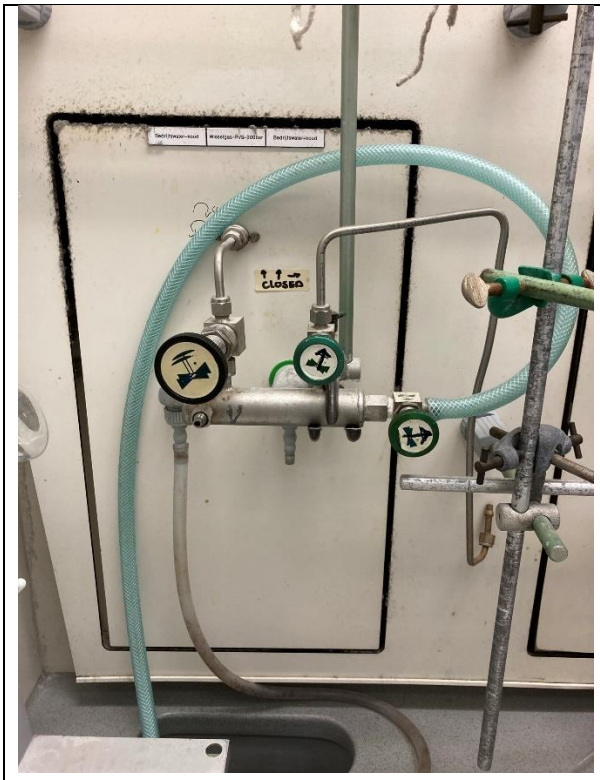

Figure 11: Autoclave filling setup in fumehood 03.115-H9

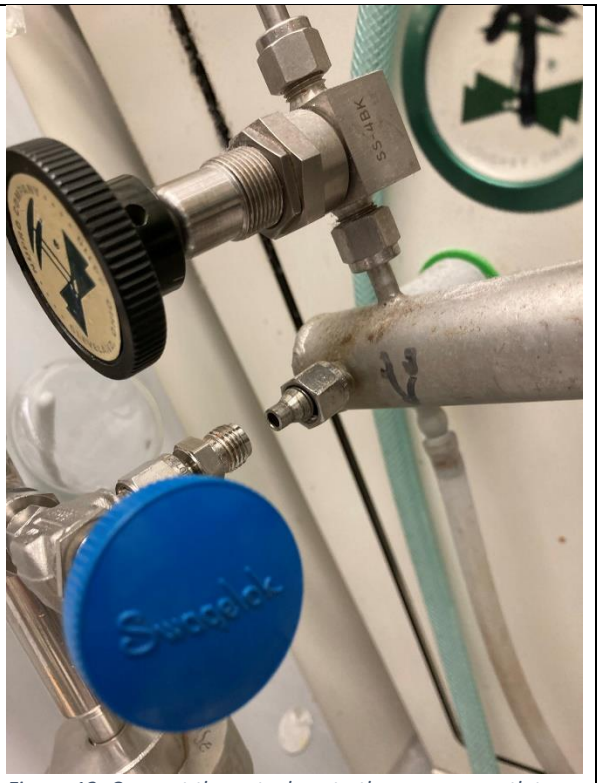

Figure 12: Connect the autoclave to the pressure outlet

Close the valve tightly with the spanner, that is marked with a red sticker You can find it in the general tool box placed of lab 03.115. This is the only screw that does not follow the “righty thighy, lefty loosey”-rule

Figure 13: Spanner that is marked with a red sticker

Figure 14: After the closing it should look like this

What follows now is a consecutive opening and closing of various valves. To be more concise, I will use a numbering indicated as follows.

Figure 15: Numbering of valves in the gas cylinder cabinet (HG03-100 GC2)

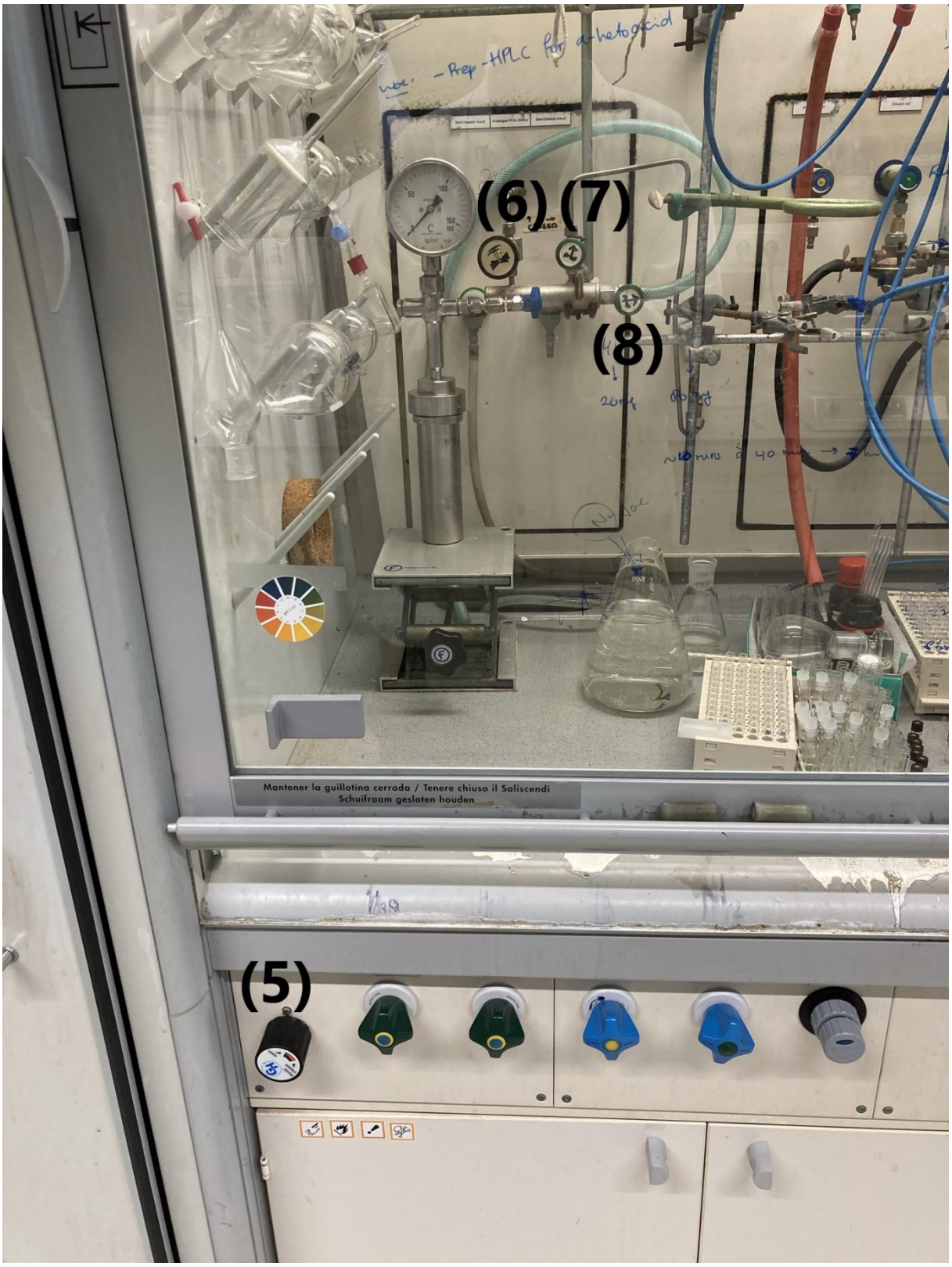

Figure 16: Numbering of the valves in fumhood 03.115-H9

First make sure that **all** (also the ones in the fumehood!!!) valves are closed (except for the valve above the gas cabinet right next to the ventilation pipe).

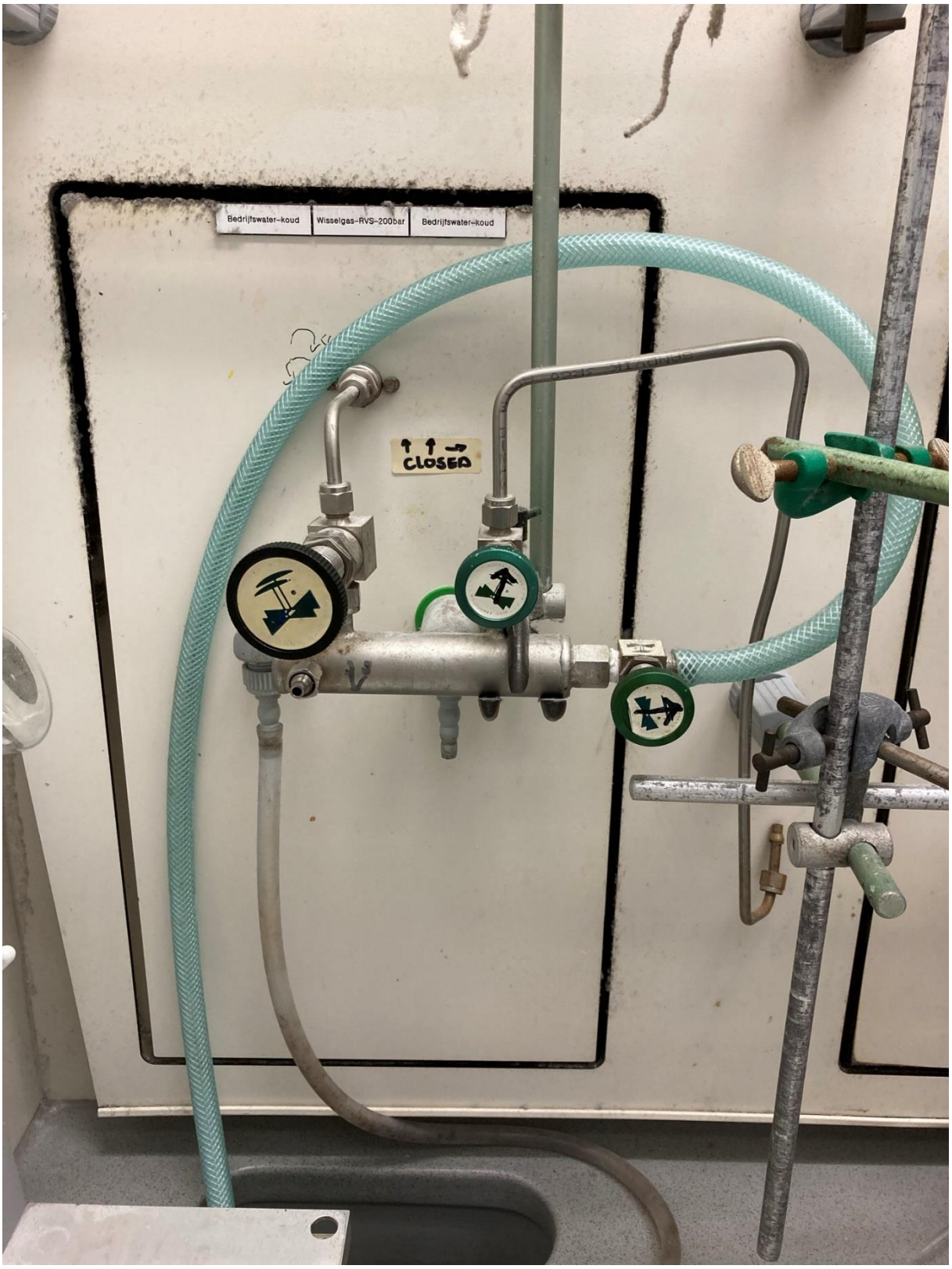

Figure 17: This is the closed state of the valves 6-8. The scheme in the background indicates the closed state in reference to the arrows (note: vlave (7) and (8) lead into an open end

Go to the gas cabinet (HG03-100 GC 2) and connect the metal hose to the pressure regulator on the flask. Pay attention that the metal ring snaps into place securing the hose tightly to the pressure regulator.

Figure 18: Metal hose connected to the gas cylinder

Then, open the main valve (1) on the pressured flask. You should see now that the first barometer shows the pressure of the flask. Now you have to turn the valve on the pressure regulator (valve (2)) (note: you will find the hand screw just hanging there loosely. This is the closed state) until you have reached the desired pressure. Do not go beyond the red line as this is the maximum the pressure regulator can handle. Valve (2) is the only valve that should not be turned completely open/closed!!!

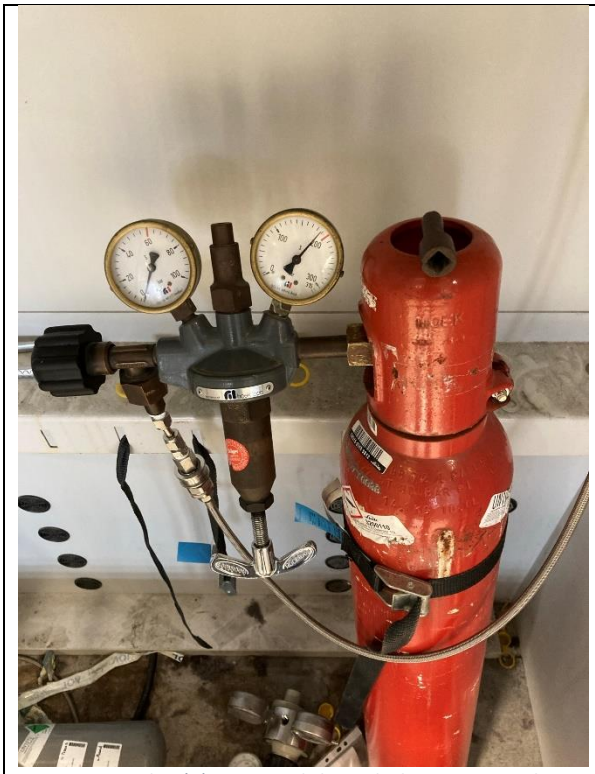

Figure 19: Valve (1) is opened the right barometer indicates the pressure present in the gas cylinder

Figure 20: Valve (2) is opened until the desired pressure is reached (in this case 60 bars)

Then open valve (3), then open valve (4), you should be able to observed the same pressure as on the regulator above.

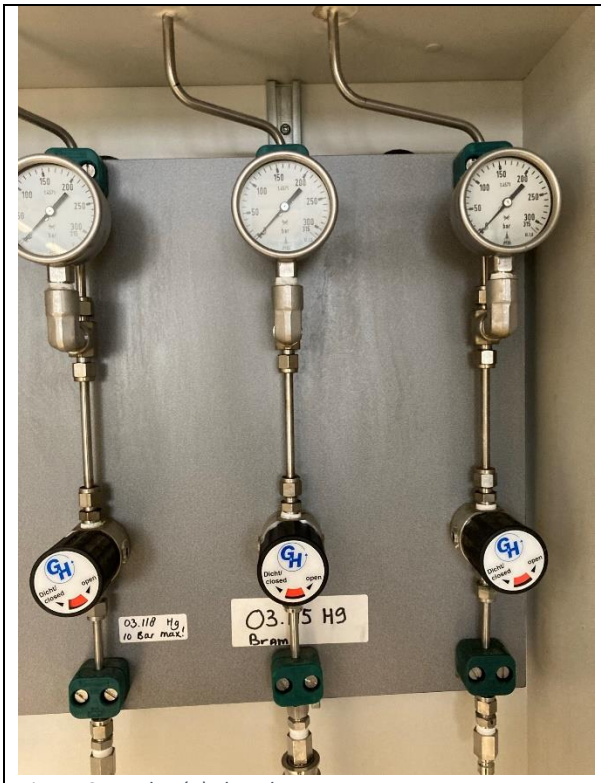

Figure 21: Valve (4) closed

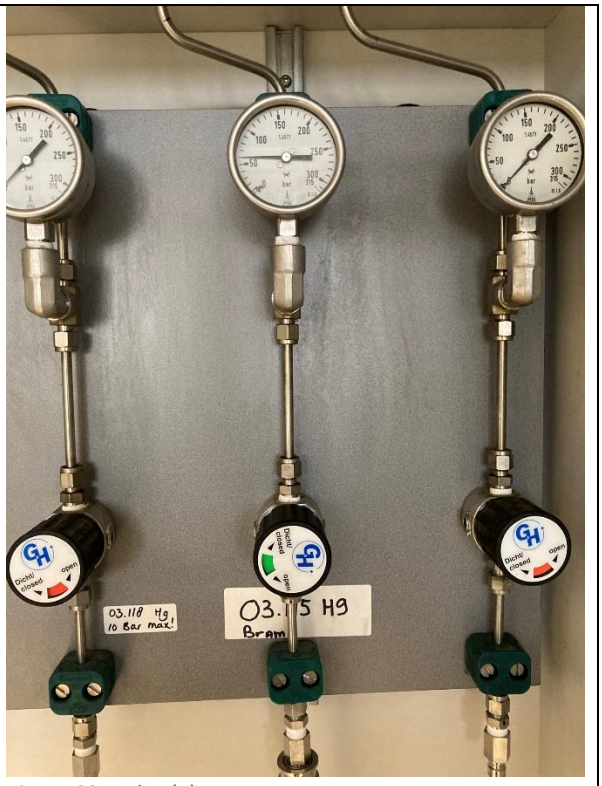

Figure 22: Valve (4) open

Now go back inside of the lab and open valve (5). The pressure goes almost into the autoclave filling device. Open the black valve (6). The gas now fills the small cylinder to which valves 6-8 and the autoclave is connected. Close the black valve (6) and open **one** of the green ones (valve (7) or valve (8)) to flush the small cylinder with your desired gas. Close the green valve and repeat the process 2 more times. (Note: it is very important that you **NEVER** open a green and the black valve at the same time, because then the whole line is open and your cylinder empties within seconds)

Now open the blue valve on the autoclave and then slowly open the black valve (6) to fill the autoclave (you can also do it the other way around, but I find it easier to dose the gas inflow with the black valve). You see the pressure of the barometer slowly rising. Close the black valve (6) and open one of the green valves (valve (7) or valve (8)) to empty the cylinder again. Close the green valve and repeat 2 more times to flush the autoclave with your desired gas.

Then close the blue valve and go to the gas cylinder cabinet (HG03-100 GC 2). Close the main valve of the pressure flask (1).

In order to release the remaining pressure in the gas pipes, go back to the fumehood 03.115 H9 and open the green valve slowly. Once the gas stops flowing close the remaining valves in decreasing order and once all valves are closed, detach the metal hose from the pressure regulator. (Note: As you come back to the gas cylinder cabinet, you should see that all of the barometers indicate 0 pressure)

Go back to the fumehood 03.115 H9, open one of the green valves and use the red marked spanner to detach the autoclave. (Open the green valve in the case that there is some remaining pressure. In this way it does not blow into your face while detaching the autoclave). Close the green valve again.

Thank the person working in 03.115 H9 that you were able to use their working space 😊.

You can put the autoclave on a stirring plate and even heat it if necessary. For that you need a “pan” (see picture). Put the thermometer into the prepared hole.

Figure 23: Pan with thermometer connected to the prepared hole

To terminate the reaction and release the pressure, you have to slowly open the blue valve. Then evacuate and backfill as described above and go to lab 03.113 to open the autoclave using the vice and spanner.