# User manual of the Nikhef gas filling station

23-6-2017

Fred Hartjes

1. *Installing the gas bottle in the water container*

If the bottle is not completely empty, then take the bottle outside the building and let the remaining gas escape in free air. Put the bottle in the water container and lay the black PVC push-down ring on top of it. Push the bottle down to the bottom of the container with one hand and slide the plastic ring through the guidance blocks on the vessel. Rotate the ring slightly to the right until the guidance blocks touch the stainless steel bolt heads on the ring.

1. *Making the gas connection*

Rotate the bottle to the right until the gas connection of the valve is pointing towards the slit in the water container. Push the black hose in the slit and attach the hose to the bottle (nut has left handed thread). Check if the white Delrin ring is still present on the hose. Tighten the nut with the 30 mm spanner on the board.

1. *Adjusting the water level and the bath temperature*

First install the gas bottle in the water container before switching on the Julabo water bath.

1. Open the lid of the Julabo water bath and check if the water level is no more than 1 cm under the top plate. Fill with water if needed, keep the lid off.
2. Power on the Julabo thermostat bath (red button) while keeping a filled water bottle ready. Normally the water level drops by several cm. Add water as soon as the level is more than 4 cm under the top plate. If the water level becomes too low (E 01), the Julabo pump stops and a continuous beep is heard. In this case switch off the Julabo for at least 4 s, add water making the level 1 cm under the top plate and power on the Julabo again. If the water level in the big container is low this procedure has to be repeated several times. Be careful with adding the water: too much water causes an overflow.
3. Once the water level is stable while running the Julabo, close the lid and adjust the temperature. Do this on the service panel with the ^ and ˅ buttons. Confirm by “OK”.
4. Leave the bath running during the whole filling procedure.
5. *Connecting the gas supply bottles*

Connect the gas supply bottles to the appropriate channels. Each of the three channels has its own preference for certain gases, given by the thread of the nut of the pressure regulator. Since there are different threads in use for the various gas bottles, here are three different nut threads on the regulators in use: CH1: LU1, CH2: RU3, CH3: RU1. Leave the main valve on all bottles still closed.

1. *Starting the LabVIEW control program*

Open the shortcut D:\Gas filling\Gas filling control main.vi and run the program. Basically the program fully controls the whole filling process, but will often ask you to do certain handling or checks. Often something has to be inserted in a LabVIEW control. Yellow means that you have to insert something, white means that the program fills it as a check.

* 1. Compose Mixture

We start with the tab window <Compose mixture>. First insert the number of components of the mixture you intend to make. Automatically the number of lines below is adapted. Using <list number> the gas is selected. It is easiest to choose the desired gas by systematically incrementing the list number. You don’t have to care about the sequence of the different gases at this stage; the program finds the most suited filling sequence by itself. The fraction (0-1) is filled in the corresponding control by typing it in. The value is entered by clicking mouse to a point outside the control. Do not give an <enter>. LabVIEW automatically fills in the fraction of the last component.

* 1. Prepare set-up

Select the bottle number from a drop-down list. Select the channel of the background gas. This gas will be used to flush the bottle after the first evaluation to get rid of traces of the previous mixture in the gas bottle. The program automatically dedicates the supply bottles to the most suited channel. Click YES if this is correct. Another connection scheme may be inserted after clicking NO.

* 1. Filling sequence

LabVIEW automatically shows here the most favourable filling sequence. After clicking <Not OK> another sequence may be selected. Note that this will often lead to a lower end pressure of the gas bottle.

* 1. Verify complete filling procedure

In this tab window the complete filling procedure is displayed for verification.

* 1. Evacuate

After opening the valve on the JSP bottle the vacuum pump is automatically started. The pressure plot shows the progress of the whole filling procedure. The user will be given instructions that have to be followed up meticulously.

* 1. Flush background gas

For the background gas one of the components the mixture is chosen. Traces of the background gas will be present in the final mixture. As an example, for a DME/CO2 mixture choose CO2 as a back ground gas, for an Argon/isobutane mixture choose Argon.

After evacuating, the bottle will be temporarily filled with 1 bar of the background gas and re-evacuated again to remove traces of the previous mixture.

* 1. Add the gases to the bottle

In this tab window the gas components will be added one after another. Compressing gas in the bottle will generate some heat. The heat is transferred to the water cooled walls with a time constant of 36 s. A second time constant (4.3 min) of a less prominent effect is also visible on the pressure curve, possibly caused by heating of the top of the gas bottle that is not covered by water. Therefore, once the desired pressure has been reached, the system waits for 5 min to let the gas cool down. In most cases the pressure has dropped down too much and the bottle pressure is restored by a bit more gas. Sometimes a third fill will be necessarily as well.

In case of emergency, for instance if one of the supply bottles has not sufficient pressure, the gas filling can be interrupted by pressing the yellow button. However, this will of course result in a deviating mixture. But still the label that is printed at last, indicates the actual gas composition of the filled bottle.

When using small supply bottles of gases in the liquid phase like DME or isobutane, the bottle temperature may substantially drop down during filling, resulting in a too low vapour pressure. In this case one may carefully increase the bottle temperature using a warm air blower.

* 1. FINISHED

In this phase the mixture is ready and the gas bottle has to be removed from the system. Just carefully follow the instructions of the program. The new mixture will be also logged in the log file of the used bottle.

**Note**

22-4-2014. Observed vacuum leak in filling system after renewing bottle hose

Estimated volume of hose and piping to valves: 100 mL

Pressure increase in 1600 s: ~55 mbar => 10-4 bar/s @ 100 mL => 3.4 µL/s @ NTP.

If this is air, then we would introduce in 2 min under vacuum 0.41 mL air or 0.088 ml O2. At a bottle of 12.3 L content this gives 7.1 ppM O2. Leak is possibly caused by leaks in the magnet valves.