This device allows to feed reactions with gas, at the desired constant pressure in the range 1- 10 bar(a), and monitors gas consumption along the reaction via the decrease of pressure in an internal reservoir. For pressure programing and data recording, the device must be connected via Bluetooth to a PC.

The device is sized for reaction runs consuming 0.5 mmol of gases or below.

Warnings:

The device can be charged at a **maximum IN pressure of 18 bar(a)**. If accidentally filled above this pressure, disconnect the IN quick connector and turn the 2-way valve to the setup position to release excess pressure asap. Pressures above 25 bar(a) can irreversibly damage the internal electrovalve that regulates working pressure.

Reactions above 2.5 bar(a) must be carried out in a Man on the Moon **milireactor** or another high-pressure vessel. Do not carry out reactions above 2.5 bar(a) in glass reactors.

Installation:

Man on the Moon X203 Software (windows only):

As of version X203, all our gas uptake devices use the X203.exe control program.
Download the software installer. Decompress the archive diskX203.rar and run the diskX203/setup.exe file. This installs the application X203.exe and necessary parts of the National Instruments Labview software. Accepting the proposed (default) installation folders is strongly recommended. Installation takes several minutes and requires computer restart.

Pairing the X204 device:

Each X204 device is labelled with the name that it shows to computers. Since June 2022 the kit no longer contains additional software or hardware to set the Bluetooth functionality in computers running old versions of windows. If this is your case, you should enable the BT connection by your own means. X204 pairing procedure is similar to any other Bluetooth device:

Turn on Bluetooth

Plug the X204 device and find it in your computer using the windows configuration tool: **configuration > Bluetooth and devices > Add devices ….**

On the X204 device name select: **Pair**. The access code for pairing is: 1234 (the default one)

After pairing, the computer assigns a COM# serial port to communicate with the device. You need to know this port number to run the X203 application.

Find this COM# port in: More Bluetooth options > COM port (outgoing).

Troubleshooting:

In their initial experiment, customers in the EU frequently report a lack of communication between the device and the computer. If this is the case, verify that the windows OS regional settings on the computer receiving the data assigns a period (.) to separate decimals and a comma (,) to separate thousands. Otherwise, the communication will not occur.

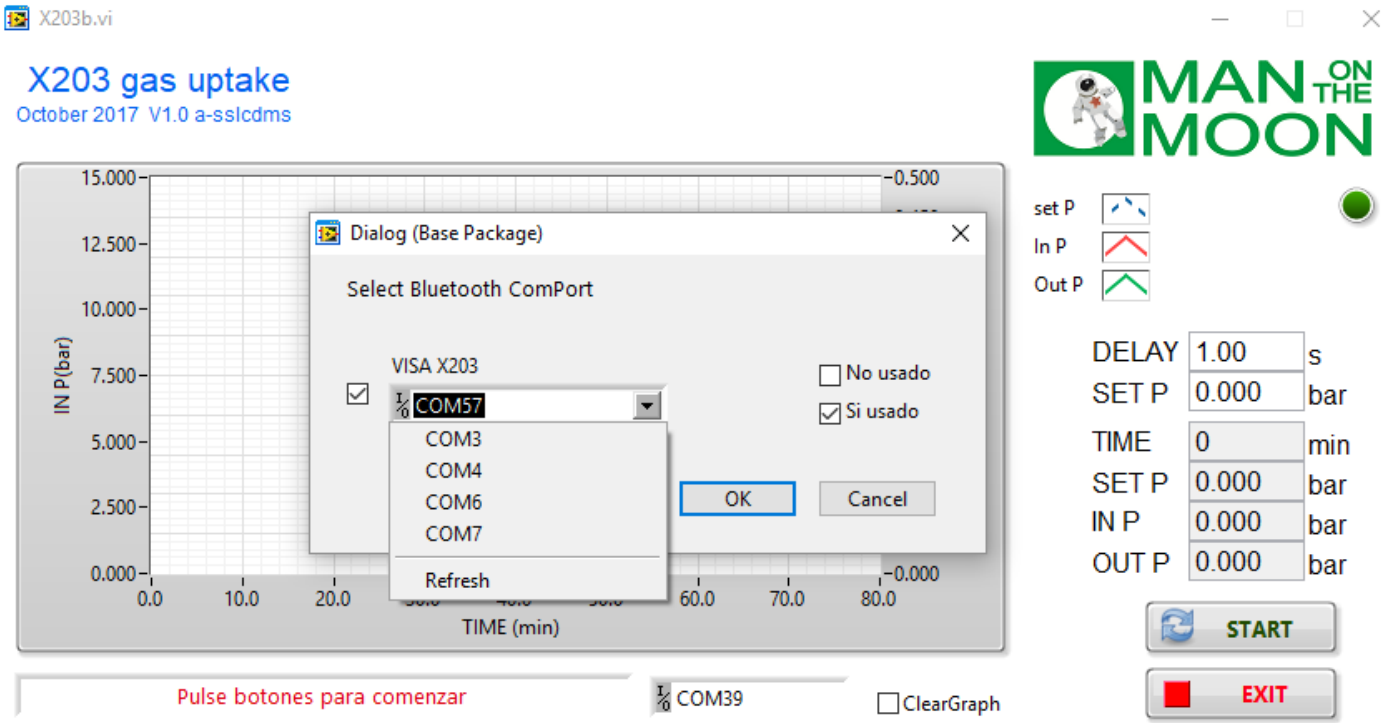
System assembly:

A picture of the assembled system is shown below. Assembly just requires three “click” connections. Once assembled, one side of the system must be connected to the high-pressure gas supply. This end consists of a ¼” O.D. flexible tubing compatible with most adapters (not included) to high pressure cylinders, etc. The other end, at the corresponding reaction vessel, either a **glass reactor** or a high pressure **milireactor,** should connect to an inert gas/vacuum (Schlenk) manifold.

The **glass reactor** connects to the switchable 3-way valve via a *Torion screw* and a piece of Nylon (polyamide) or PTFE tubing of 5 mm O.D. This *screw* and the *septum cap* of the flask must be carefully tightened and checked before every experiment. The 3-way valve can be switched between two positions (180°). In one position the **glass reactor** connects to the exterior, so that it can be used as a conventional Schlenk flask in a vacuum/inert gas manifold to facilitate sample preparation. In the opposite position the flask connects to the control unit to be filled with gas at the desired pressure. Positioning the valve between the two positions (90°) disconnects the flask from both the Schlenk manifold and the control unit. The **milireactor** can be used essentially in the same way as the glass reactor, but it has its own instructions.

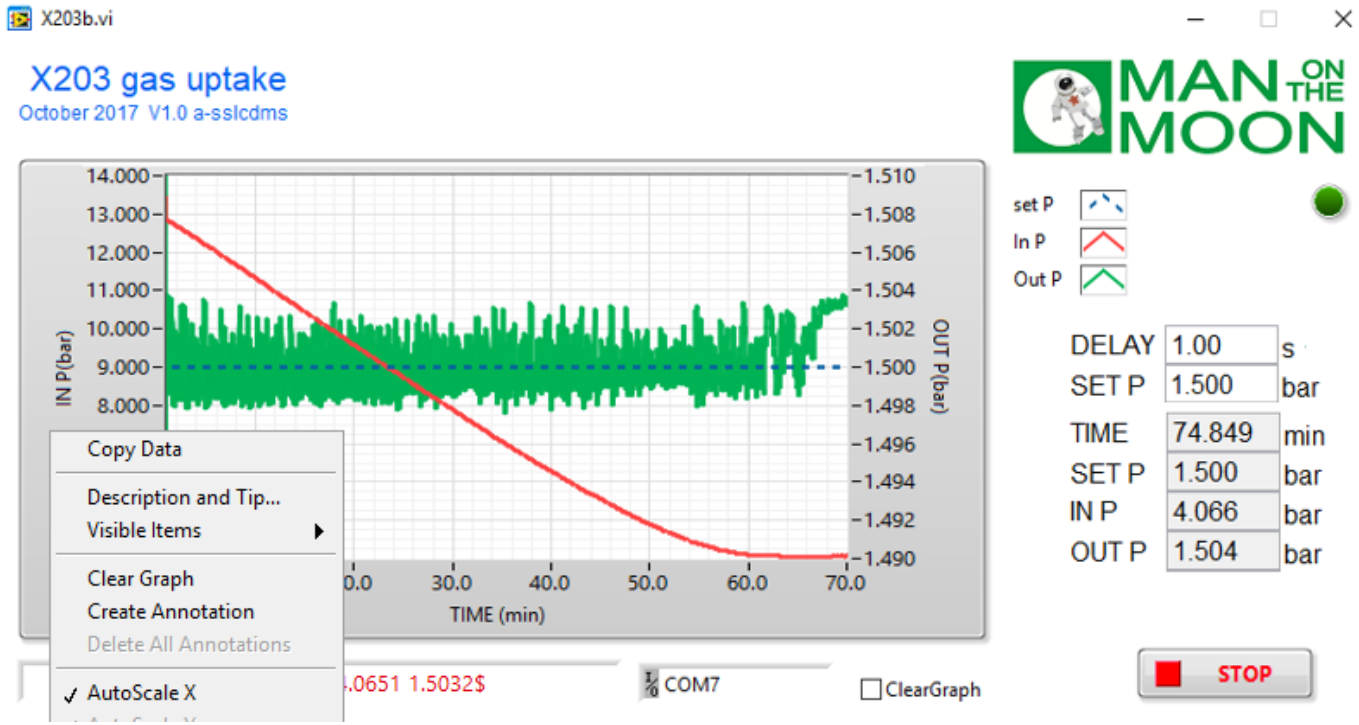
Running the X203 application:

Open the X203 application. First, it asks for a communication port (COM#). Select that assigned to the current X203 device or click in the refresh option if not in the list.



Once the application starts receiving data, they can be stored in a .dat ASCII file clicking in the **START** button. This opens a dialog window to name and locate the data file as desired. Each DELAY interval (in seconds), the program writes the total reaction TIME (in minutes), the reservoir absolute pressure (IN), the programmed absolute reaction pressure (SET P) and the real reaction pressure (OUT) (in bar). The DELAY interval and the SET P can be modified anytime by overwriting the previously entered value. The .dat file can be opened with excel and many other programs. Note that the application interface displays the reservoir pressure (IN) and the reaction pressure (OUT and SET P) in different Y axes.

The option **ClearGraph** refreshes the screen but does not erase the data already collected in the .dat file. Clicking the mouse (right) button in certain areas displays different options menus. When the **AutoScale** options are disabled, the scales can be modified as desired overwriting the numerical values at the ends of the axes. The **STOP** button stops the current data acquisition, and **EXIT** closes the application. Note that none of these commands requests the control unit to change the SET P from the last entered value.



Comments on operation:

Even when the X203 application is not running in the computer, the (plugged) control unit displays the actual IN and OUT pressures, the last entered SET P (zero by default), and the current value of the electrovalve opening (only relevant for programming and service). Visualizing such information during connection of the control unit to the high-pressure gas source helps to avoid overpressure, and also may assist the disassembling of the system and the releasing of pressure after reaction monitoring. To release pressure from **milireactors**, please consult the instructions.

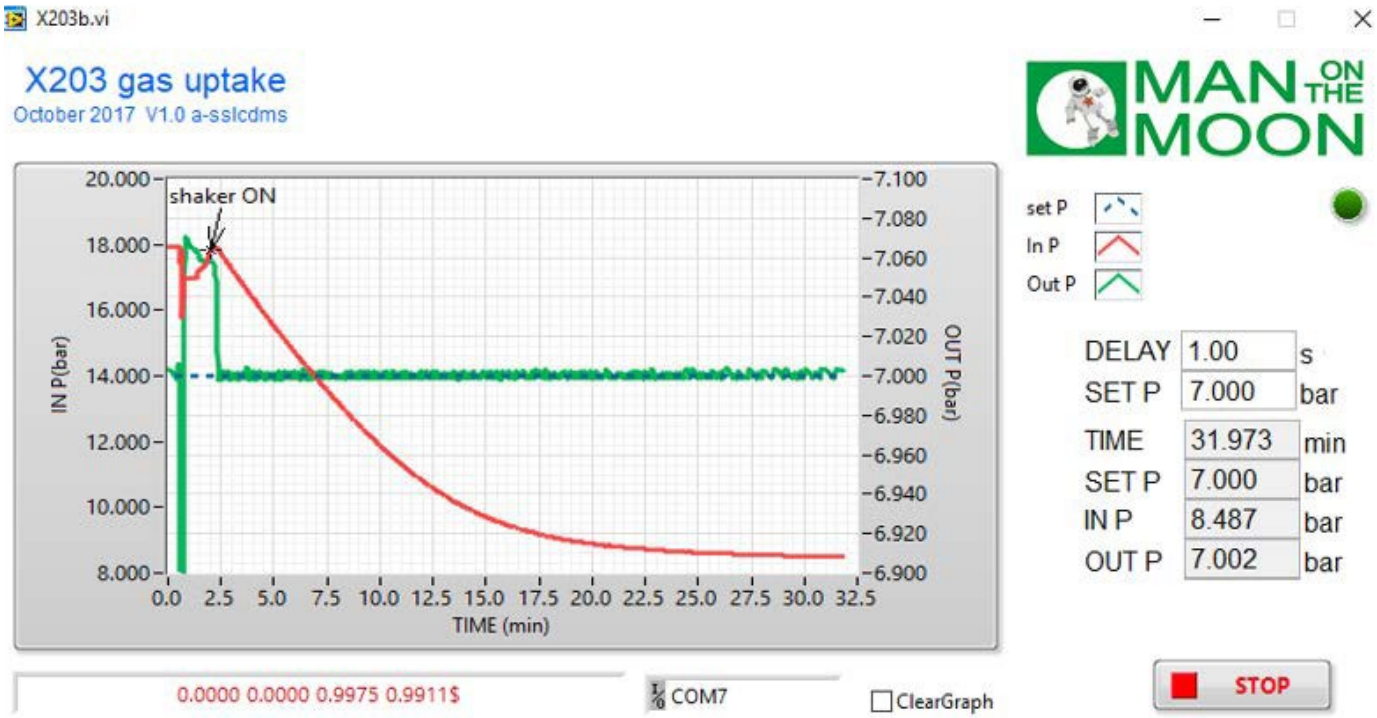
Before operation the device may need to be cleaned from the gases of previous operations. Vacuum may be applied to clean **glass reactors** or **milireactors**, but to purge the rest of the system (from the source of high pressure to the switchable 3-way valve) it is safer to pass a controlled stream of gas. For example, set up a dummy experiment with SET P 1.2 bar and open the reaction vessel until the reservoir empties, repeating the procedure at least three times.

Reaction setup:

Although the DELAY interval and the SET P can be modified anytime during data collection, the control unit can only respond if prompted to increase pressure, because the only way to release pressure from the reaction is manually. Therefore, reaction setups should be conceived to reach the working pressure (SET P) from below.

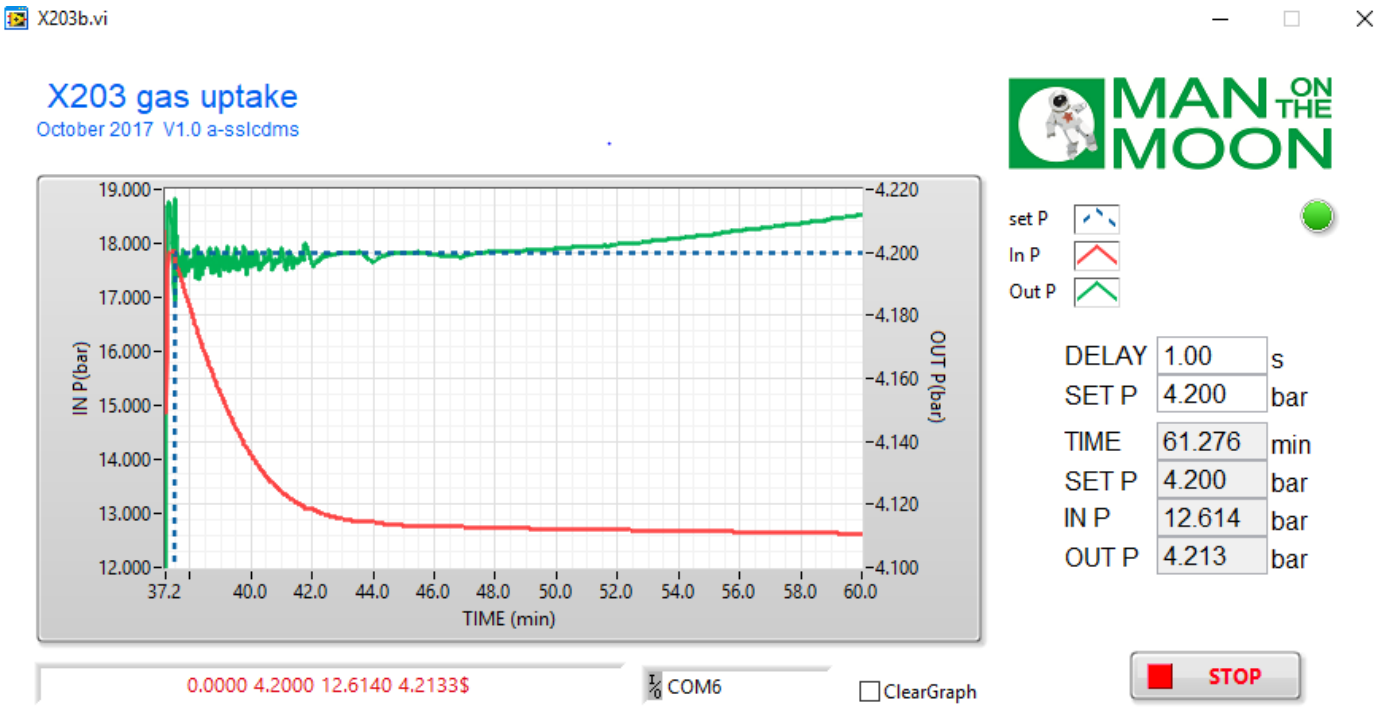
The switchable 2-way valve of the control unit connects or disconnects the internal reservoir of the device from the high pressure supply. Hence, it should be switched to close the reservoir after reaching the desired reaction pressure (SET P) (reaction time t=0). Afterwards, the device should be able to maintain the reaction pressure (OUT) within an interval of +/- 5 mbar around the SET P, at the expense of a pressure decrease in the reservoir that portraits reaction kinetics.

Depending on the actual reaction setup procedure, the initial approach to the desired reaction pressure (SET P) may not be perfect, provoking undesired overpressures in the reaction vessel. Typically, such overpressures should not exceed a few mbar but, occasionally, they may be as large as 100 mbar. In the interval of reaction pressure from 1 to 2.5 bar, these initial overpressures can be quickly released through the septum of the **glass reactor** (with the help of a microsrynge needle for example), although when using the **milireactor** such corrections are not that easy. The initial overpressure will cause a lag period in the gas consumption curve, since the electrovalve shall not open the reservoir until the excess gas is reacted. As an example, the figure below displays the course of a catalytic hydrogenation using the **milireactor**. Note that the initial fill of the reactor is wrong by about 60 mbar but, as soon as reaction is initiated by turning on the shaker, the overpressure is readily consumed and its effect in the reaction profile is nearly irrelevant. Yet, the sizing of the reactions and the strategies for reaction setup should take into account the possibility of these initial lag periods. Instead of extrapolate the lag period or manually release the overpressure, you may also consider to quickly change the SET P value to the actual OUT P, to immediately take control, whenever you do not need to match an exact value of the working pressure.



Reaction finish:

Importantly, the internal electrovalve of the control unit is not able to close hermetically. As a consequence, there is always a residual flow from the reservoir to the reaction. The magnitude of such a “leak through” can be rather variable since it depends on the actual pressure difference between reservoir and reaction, and may change each time the electrovalve opens/closes. Its impact during setup and the course of reactions is negligible, but it may blur the end of reactions since baseline will never become totally horizontal when reservoir is closed (run position). Still, the end of the reaction should be evident attending to the reaction pressure, which goes out of control because the incoming gas cannot be either consumed or released. The example provided below corresponds to a catalytic hydrogenation run of cyclooctene at 4.2 bar. Reaction pressure (green) irreversibly surpass the SET P when reaction finishes. Note that the volumes of the reservoir and the reaction are very different. In the small volume of the closed reservoir, the residual pressure drop at the end of this experiment is around 20 mbar/min.



Reservoir size:

Although the volume of the reservoir may slightly change from one device to another, each 0.1 mmol of consumed (ideal) gas should cause a pressure drop around 2.3 bar. If necessary, additional, more precise, calibrations of the volume of the gas reservoir should be carried out by the final user. Note that reactions with large amounts of substrates may require the refill of the reservoir during the run.

Consumables:

Septa: 12.9 mm, silicon/PTFE, 3.2 mm for GL14. VWR Ref: 548-0480
Sealing rings for Torion screw: SVL sparing sealing rings 4.8 – 5.2 mm OD. VWR Ref: BIBB701-20
Flexible Nylon (polyamide) tubing - 5mm OD X 3mm ID
Nylon cap for GL14 screw. Sigma - Aldrich Ref. Z680567