



This apparatus facilitates the introduction of gas into reactions, maintaining constant pressure within the specified range of 1-10 bar(a). Simultaneously, it tracks gas consumption throughout the reaction by monitoring the pressure decrease within an internal reservoir.

To enable pressure programming and data recording, the device requires Bluetooth connection to a PC.

The device is optimized for reaction runs involving gas consumptions of 0.5 mmol or lower.

Cautions:

The device should only be charged with a **maximum input (IN) pressure of 18 bar(a)**. If inadvertently filled beyond this limit, promptly disconnect the IN quick connector and switch the 2-way valve to the “*setup*” position to release excess pressure. IN pressures exceeding 25 bar(a) may result in irreversible damage to the internal electrovalve responsible for gas dosing.

Reactions surpassing 2.5 bar(a) should be conducted using a specialized high-pressure vessel such as the Man on the Moon **milireactor**. **Avoid performing reactions above 2.5 bar(a) in glass reactors**, as they are not designed to withstand such elevated pressures.

Exercise caution and adhere to these guidelines to ensure the safe and effective operation of the equipment.

Installation:

Man on the Moon X206 Software (Windows OS only):

Download the [software installer](#). Decompress the (zip) folder [diskX206](#) and run the [X206/Volume/setup.exe](#) file. This installs the application [X206.exe](#) together with the 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 X206 device:

Starting from June 2022, the kit no longer includes additional software or hardware for configuring Bluetooth functionality on computers running old versions of the Windows OS. If you fall into this category, it is necessary to enable the Bluetooth connection using your own methods.

The X206 pairing process is akin to that of any other Bluetooth device:

Activate Bluetooth on your computer.

Plug in or connect the X206. When the display appears, the device name briefly shows in the format M5StickC_XXXY. Locate this device name on your computer using the Windows configuration tool:

Configuration > Bluetooth and Devices > Add Devices ...

Click on the X206 device name and then select "**Pair**".

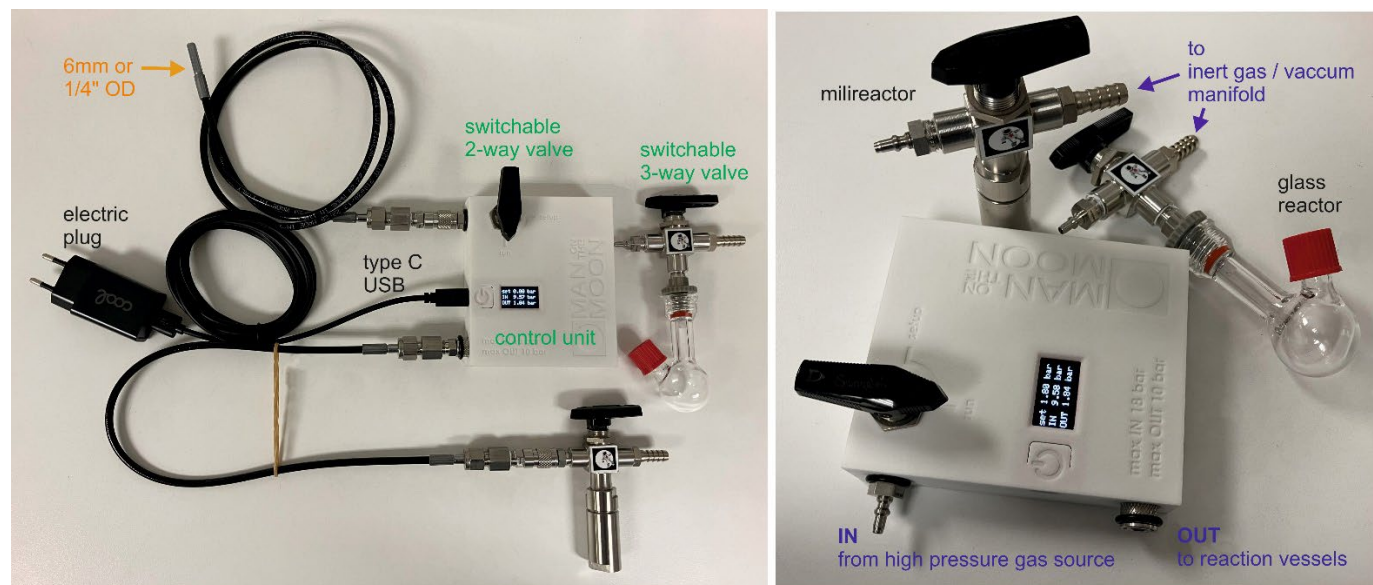
Upon successful pairing, the computer designates a COM# serial port for communication with the device. Take note of this port number as it is required to operate the [X206.exe](#) application. Locate the COM# port under: **More Bluetooth Options > COM Port (Outgoing)**.

Troubleshooting:

Even after successful installation and pairing, EU users often report lack of communication between the device and the computer. In such instances, it is crucial to confirm that the Windows OS *regional settings* on the computer receiving the data utilize a period (.) to denote decimal points and a comma (,) to separate thousands. Other settings cause communication failure.

System assembly:

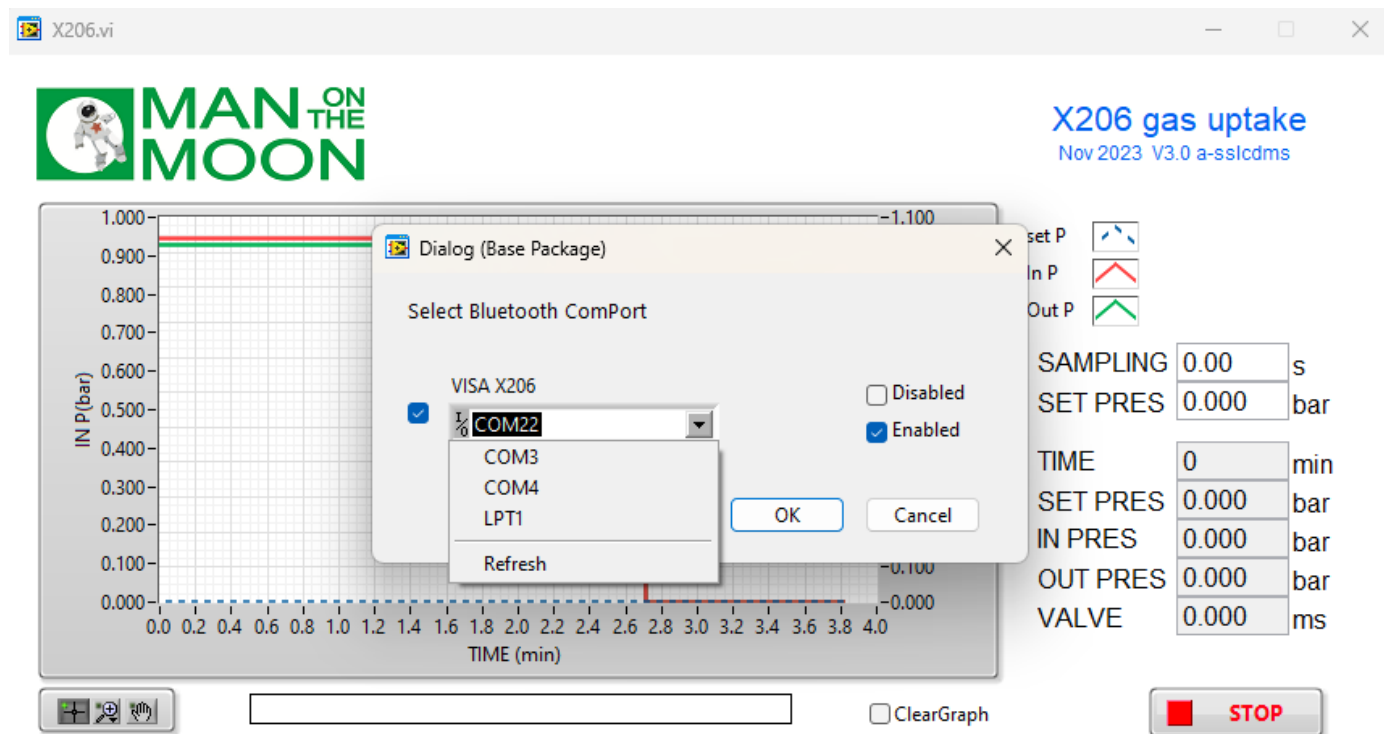
A picture of the assembled system is shown below. Assembly just requires three “click” connections. Once assembled, one side of the system must be connected to the high-pressure gas supply. This end consists of a **6mm (¼”) O.D. flexible tubing** compatible with most adapters (not included) to high pressure 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 outside, 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 X206 application:

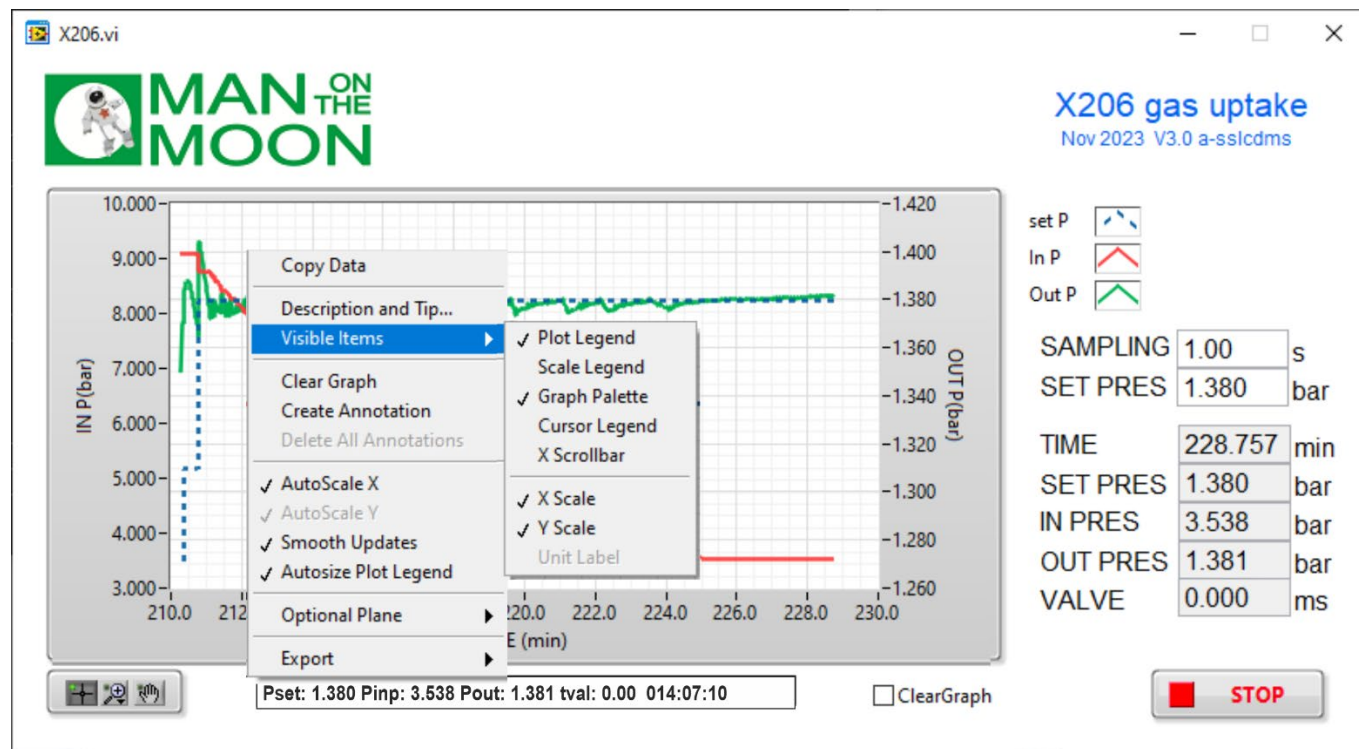
Open the [X206](#) application. First, it asks for a communication port (COM#). Select that assigned to the current X206 device or click in the [refresh](#) option if not in the list.



Clicking in the **START** button opens a dialog window to name and locate the file to store data, a .dat ASCII file. Each SAMPLING interval (in seconds), the program writes the total reaction TIME (in minutes), the reservoir pressure (IN PRES), the programmed absolute reaction pressure (SET PRES) and the real reaction pressure (OUT PRES). All pressures are absolute pressure in bar. The data file also stores the current aperture value (ms) for the electrovalve (VALVE) that doses the gas from the reservoir to the reaction.

The SAMPLING interval and the SET PRES 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) in opposite Y axes.

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



Comments on operation:

Even when the X206 application is inactive on the computer, the control unit will still display the actual IN and OUT pressures and the last entered SET PRES (zero by default). Observing this information while connecting the control unit to the high-pressure gas source helps to prevent IN overpressure. It also aids in system disassembly and pressure release post-reaction. Refer to the [instructions](#) for proper pressure release from milireactors.

Before operation, the device may need to be cleaned from residual gases from previous operations. While vacuum may be suitable for cleaning **glass reactors** or **millireactors**, purging the entire system (from the source of high pressure to the **switchable 3-way valve**) is safer using a controlled gas stream. For instance, conduct a dummy experiment with SET PRES 1.2 bar, open the reaction vessel until the reservoir empties, and repeat the process at least three times.

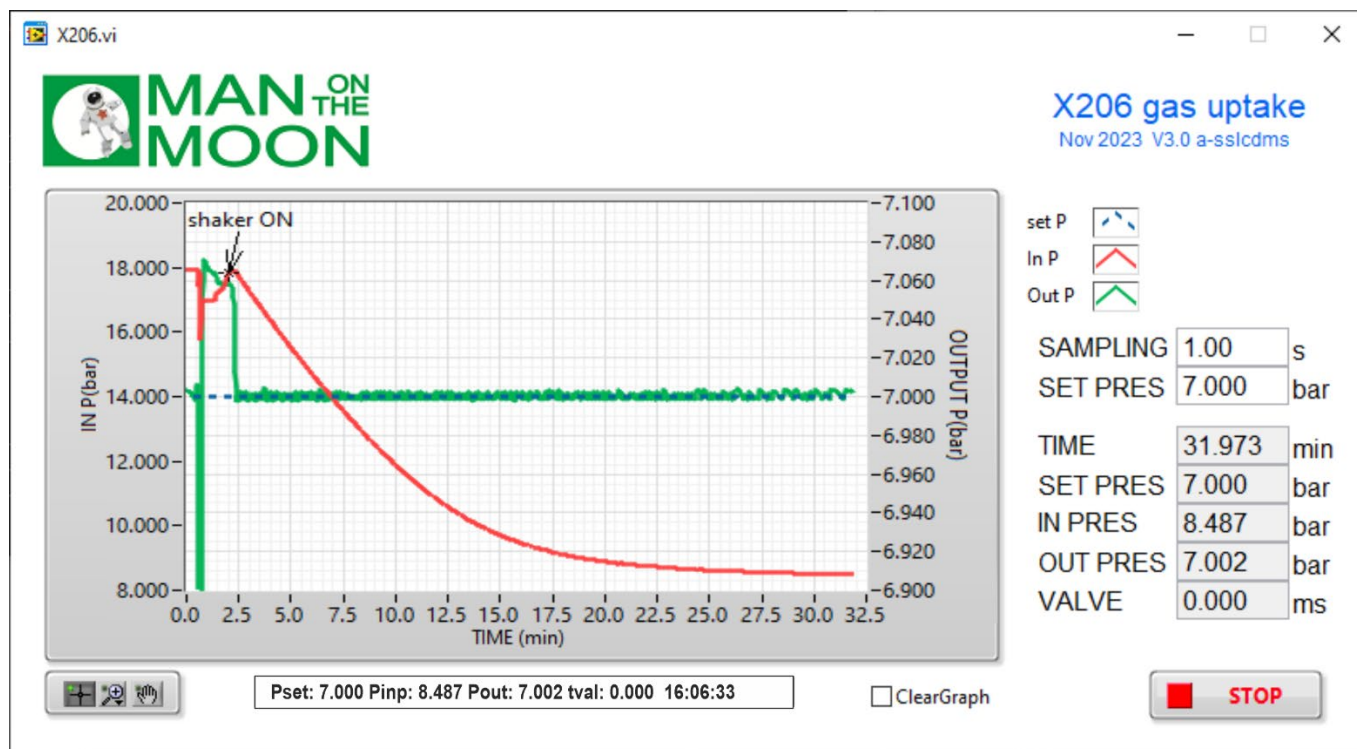
If transferring the **control unit** into a *glovebox*, pre-clean it with inert gas as explained above. Place it in the antechamber with the power off and the **switchable 2-way valve** in the "run" position to shield delicate internal components from exposure to vacuum.

Reaction setup:

While the desired reaction pressure (SET PRES) can be modified at any time during data collection, the control unit can only respond when requested to increase the pressure, since releasing the pressure requires manual intervention. Therefore, it is essential to design reaction setups that approximate SET PRES from below.

Approaching SET PRES too rapidly may result in overshooting the intended value. To mitigate this potential issue, the gas-dosing electrovalve is programmed with a restricted maximum aperture. Because of it, if SET PRES value is high, approaching it in the (typically evacuated) reaction vessel can take quite a while, severely obscuring the initial part of the reaction. To expedite this process, it is advisable to pre-fill the line preceding the vessel's **switchable 3-way valve** with overpressure. This ensures that when the valve opens for the reaction, the pressure swiftly reaches the proximity of SET PRES, requiring only a final adjustment by the electrovalve. To implement this, temporarily elevate SET PRES to the estimated overpressure value, allow it to be reached, and then readjust SET PRES to the intended reaction pressure.

If the initially planned pressure is nevertheless exceeded, it can be readily corrected to the intended value in **glass reactors**, for instance, by using a needle through the septum. However, such a correction is unfeasible in **millireactors**. In this case, it is recommended to promptly modify the SET PRES value to align with the current OUT PRES, ensuring immediate control. Otherwise, the initial overpressure causes a lag period in the gas consumption curve, as the electrovalve does not open the reservoir until the excess gas is reacted. In the presented catalytic hydrogenation example, the **millireactor's** initial fill is off by about 60 mbar. Once the shaker initiates the reaction, the overpressure is rapidly consumed, rendering its impact on the reaction profile negligible. However, when planning reactions in general, it is crucial to consider the potential for these initial lag periods. Achieving precise working pressures requires a thorough understanding of the actual configuration, reaction volumes, electrovalve responses, and proficiency in executing the setup procedures and the aforementioned tricks.

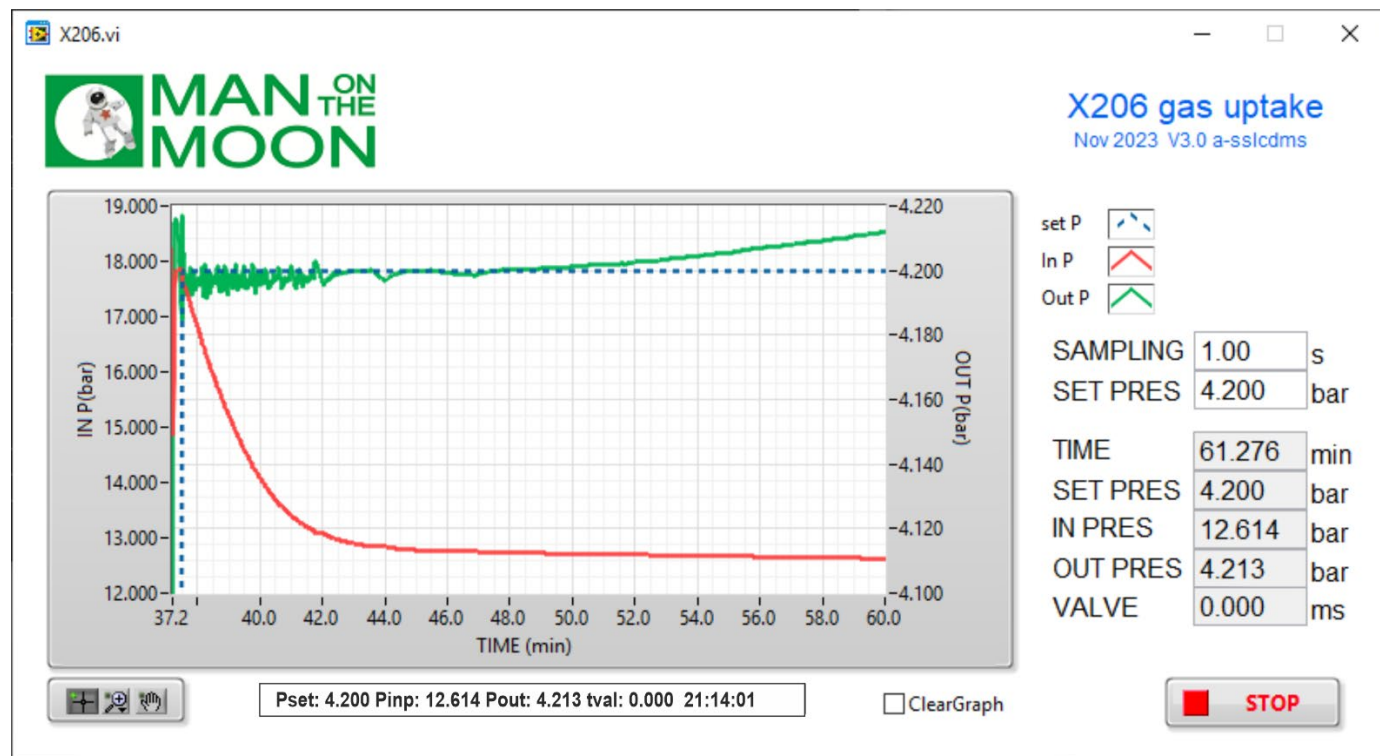


The **switchable 2-way valve** in the **control unit** connects or disconnects the internal reservoir from the high-pressure supply. It must be closed (position "run") immediately after reaching the desired SET PRES. Subsequently, the device will maintain the reaction pressure (OUT PRES) within an approximate range of +/- 5 mbar around SET PRES, which will cause a decrease in the pressure of the internal reservoir reflecting reaction kinetics.

Reaction finish:

The gas-dosing internal electrovalve of the **control unit** is incapable of achieving a hermetic seal under any condition. Consequently, depending on the pressure difference between the reservoir and the reaction, a residual flow or "leak through" may occur. Additionally, this flow may vary with each opening or closing of the electrovalve. While this phenomenon has a negligible impact during setup and the progression of reactions, it can slightly obscure the conclusion of certain reactions, as the baseline never becomes completely horizontal. Nevertheless, the end of the reaction is indicated by OUT PRES, which deviates from control since the incoming gas cannot be consumed or released. The provided example illustrates a catalytic hydrogenation of cyclooctene at 4.2 bar, where OUT PRES

irreversibly exceeds the SET PRES when the reaction concludes. In the confined volume of the closed reservoir, the residual pressure drop rate at the end of this experiment is approximately 20 mbar/min.



To minimize this issue, plan your reactions to conclude at a reservoir pressure (IN PRES) only moderately above the SET PRES. Although the volume of the reservoir may slightly vary from one X206 device to another, each 0.1 mmol of consumed (ideal) gas causes an IN PRES drop of around 2.3 bar. If necessary, additional, more precise calibrations of the gas reservoir volume should be conducted by the end user.

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](#)