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## Series X104 kit *gas evolution*

This device monitors the variation of pressure inside a closed reaction flask. The hardware and software included in the kit allow wireless data recording from a PC.

### Warnings:

Even though the measuring range of the pressure sensor is 0 – 4 bar (gauge, relative), the **pressure** during the experiment **should not exceed 1.5 bar** because of the specifications of glassware and connections. Carefully calculate the amounts of reagents to avoid exceeding this limit. The approximate total volume of the system is 15 mL when using the default reaction vessel (see below).

Prolonged contact with **chlorinated solvents** may **damage** the **housing** of the pressure sensor & data transmitter.

### Installation:

#### Man on the Moon X104 software (Windows OS only):

Download the [software installer](#). Decompress the .zip archive and run the [diskX104/X104\\_v2BT17Installer/Volume/setup.exe](#) file. This installs the application and 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. Copy or move the executable file [diskX104/X104\\_v2BT17Installer/X104v2BT17.exe](#) to the desktop or desired folder.

#### Pairing the X104 device:

Since June 2022 our kits no longer contain 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. X104 pairing procedure is similar to any other Bluetooth device:

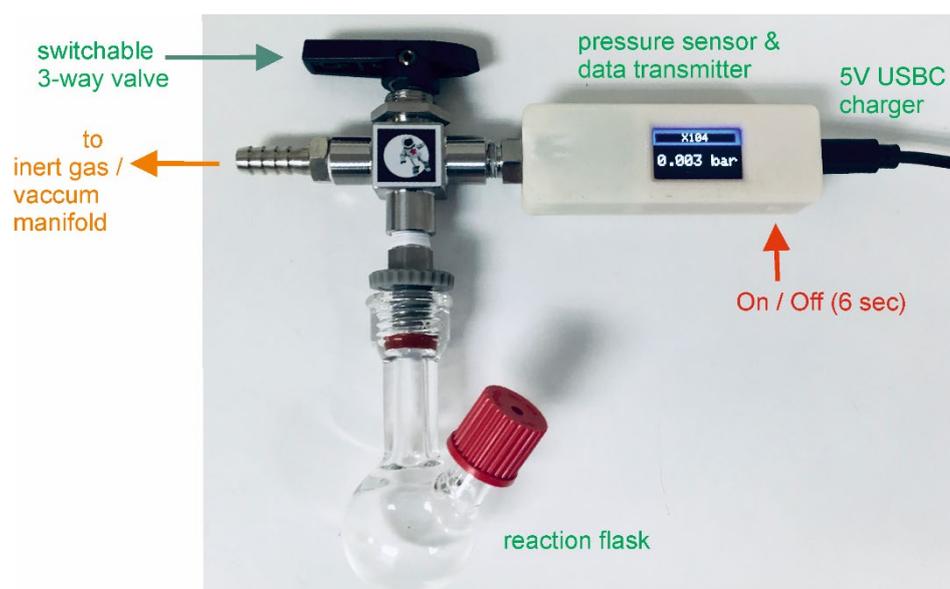
Turn on Bluetooth on your computer.

Plug in or connect the X104. Before the display shows the pressure, it shows for a few seconds the name of the device, which is in the format M5StickC\_XXXY. Find this device name on your computer using the Windows configuration tool: **configuration > Bluetooth and devices > Add devices ....** Click on the X104 device name and then: **Pair**.

After pairing, the computer assigns a COM# serial port to communicate with the device. You need to know this port number to run the [X104v2BT](#) application. Find this COM# port under: More Bluetooth options > COM port (outgoing).

### System assembly:

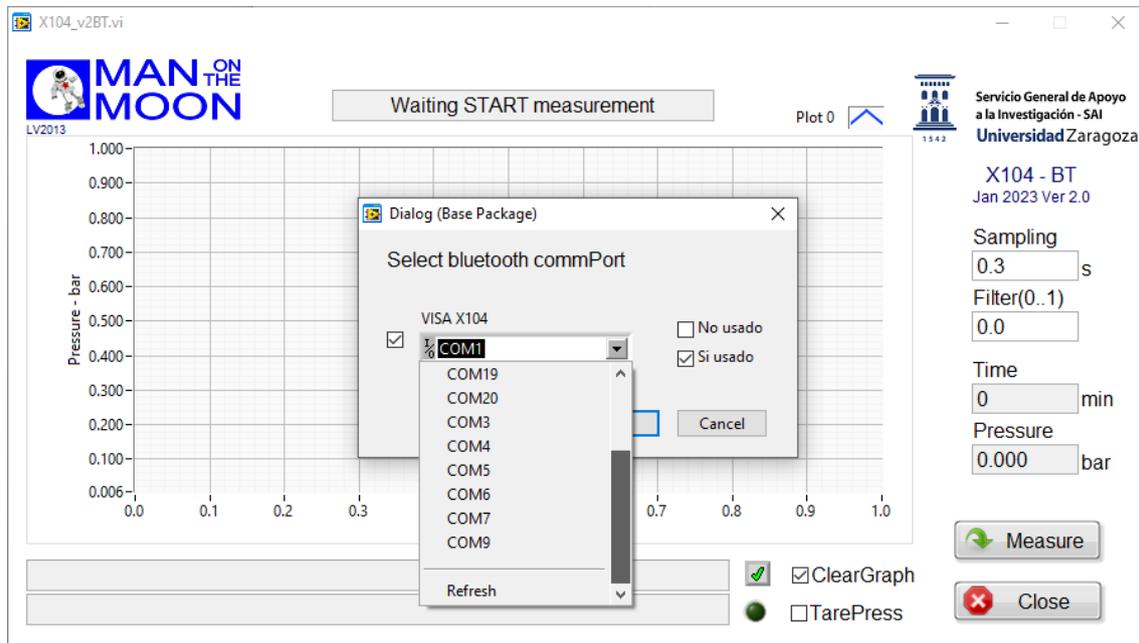
The reaction flask has to be connected to the switchable 3-way valve via the Torion screw of the flask, through a piece of Nylon (polyamide) or PTFE tubing of 5 mm O.D. This screw and the septum cap of the flask must be carefully tightened and checked before every experiment. The 3-way valve can be switched between two positions (180°). In one position the flask 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 pressure sensor & data transmitter, therefore the system is closed. Positioning the valve between the two positions (90°) disconnects the flask from both the Schlenk manifold and the sensor.



The pressure sensor & data transmitter is turned on by plugging in the USB-C charger or by connecting the internal battery by pressing the On/Off button (disconnection requires 6 seconds pressing). The internal battery has a maximum autonomy of approx. 40 min. Note that this will not be sufficient for most experiments. It is recommended to keep the device plugged in during the entire experiments.

## Running the X104v2BT application:

Open the [X104v2BT](#) application. First, it asks for a communication port (COM#). Select that assigned to the current X104 device or click in the [refresh](#) option if not in the list.

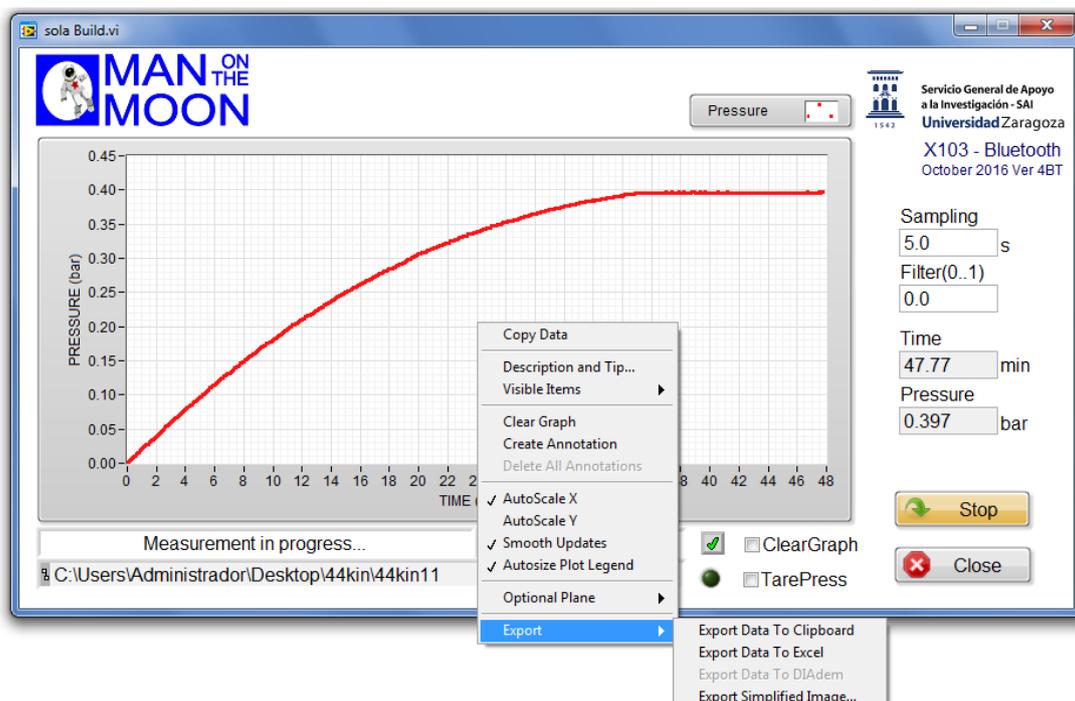


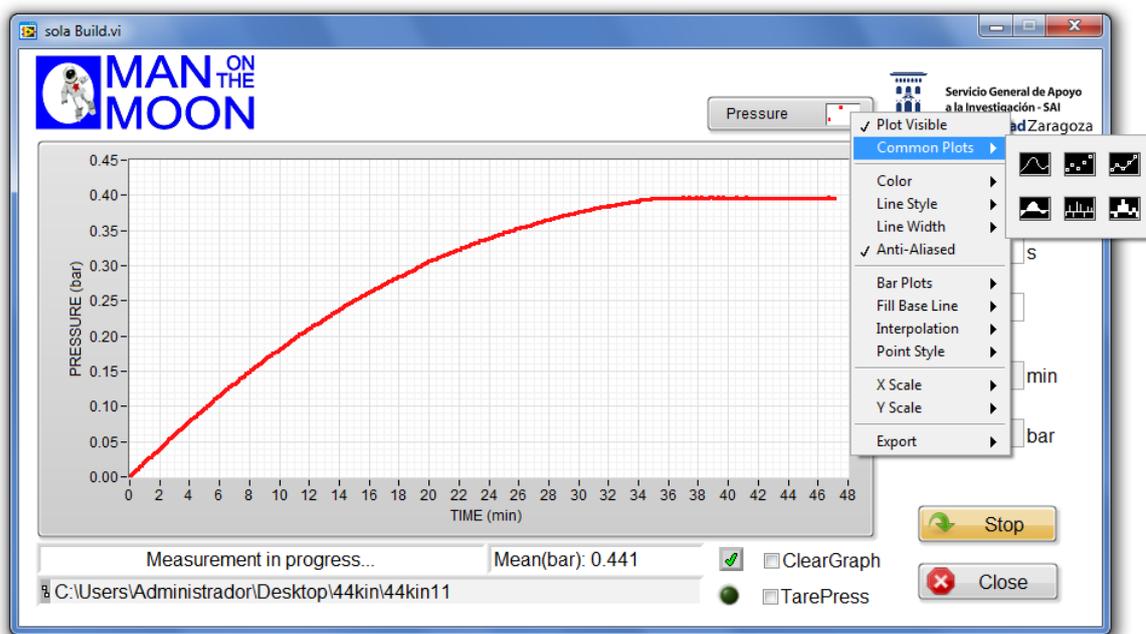
Once the application starts receiving data, they can be stored in a .dat ASCII file clicking in the **Measure** button. This opens a dialog window to name and locate the data file as desired. Each **Sampling** interval, the program writes the total reaction *Time (min)* and the *Pressure (bar)*. The .dat file can be opened with excel and many other programs.

The **Sampling** interval (in seconds) can be modified anytime overwriting the current value.

A **Filter** function can be applied to reduce noise, if necessary (**Filter** = 0 means no filter).

$$P = \text{Filter} \times (\text{previous } P \text{ value}) + (1 - \text{Filter}) \times (\text{current } P \text{ value})$$





The options **TarePress** and **ClearGraph** reset the current *Pressure* and the *Time* to zero, respectively, both in the application window and in the .dat file (but not the pressure shown in the display). Clicking the mouse (right) button in certain areas opens different options menus. When the **AutoScale** options are disabled, the scale can be modified as desired overwriting the numerical values at the ends of the axes. **Stop** button stops data acquisition, and **Close** closes the application.

### Comments on operation:

Gas evolution during the reactions is monitored as pressure increase. If necessary, calibration of the reactor total volume to transform pressure increase (bar) into conversion (mmol) should be carried out by the final user. As a reference, the previous Figures correspond to the evolution of a total of 0.30 mmol of O<sub>2</sub> from a reaction in 3 mL of solvent (water).

Please note that there is an empty space between the 3-way valve and the pressure sensor that may need to be cleaned before each experiment to remove gases from previous operations. Connect the system to the vacuum and switch (180°) the 3-way valve several times. Then, do the same operation under inert gas. This procedure will fill the mentioned empty space with inert gas.

### 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.

### 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](#)

## Reaction vessels

Each gas evolution kit contains a glass reactor the size of which must be chosen by the customer, the smallest (approx. 15 mL volume) being the default option. 35 and 100 mL approx. volume options are also eligible. Additional or double-walled vessels (for convenient thermostating, approx. 15 mL reaction volume) can be purchased separately. Contact us for prices and availability.

