Series X104 kit
gas evolution

The device serves to monitor pressure variations within a closed reaction flask.

For automatic data recording (pressure vs. time), the device requires Bluetooth connection to a PC.

Warning:

Pressure Limitation: While the pressure sensor's measuring range spans from 0 to 4 bar (gauge, relative), the pressure should not surpass 1.5 bar during experiments. This limitation is due to the specifications of the glassware and connections involved. Prior to experimentation, calculation of reagent amounts is advised to ensure adherence to this limit. The total volume of the system, utilizing the default reaction vessel, approximates 15 mL (see below).

Installation:

Man on the Moon X104 Software (Windows OS only):

Download the software installer. Decompress the (zip) folder and run the X104_v2BT17Installer/Volume/setup.exe file. This installs 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. After, copy or move the executable file diskX104/X104_v2BT17Installer/ X104_v2BT17.exe to the desktop or desired folder.

Pairing the X104 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 X104 pairing process is akin to that of any other Bluetooth device:

Activate Bluetooth on your computer.

Plug in or connect the X104. 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 X104 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 X104_v2BT17.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.

Connect the reaction flask to the switchable 3-way valve using the *Torion screw* of the flask. Ensure the connection is made through a piece of Nylon (polyamide) or PTFE tubing with a 5 mm outer diameter. Before each experiment, carefully tighten and check both the *Torion screw* and the *septum cap* of the flask to prevent leaks. The 3-way valve is capable of switching between two positions (180 degrees). In one position, the flask is connected to the outside, allowing it to function as a conventional Schlenk flask in a vacuum/inert gas manifold for sample preparation. In the opposite position, the flask connects to the pressure sensor and data transmitter, rendering the system closed. Positioning the valve between the two positions (90 degrees) disconnects the flask from both the Schlenk manifold and the sensor.
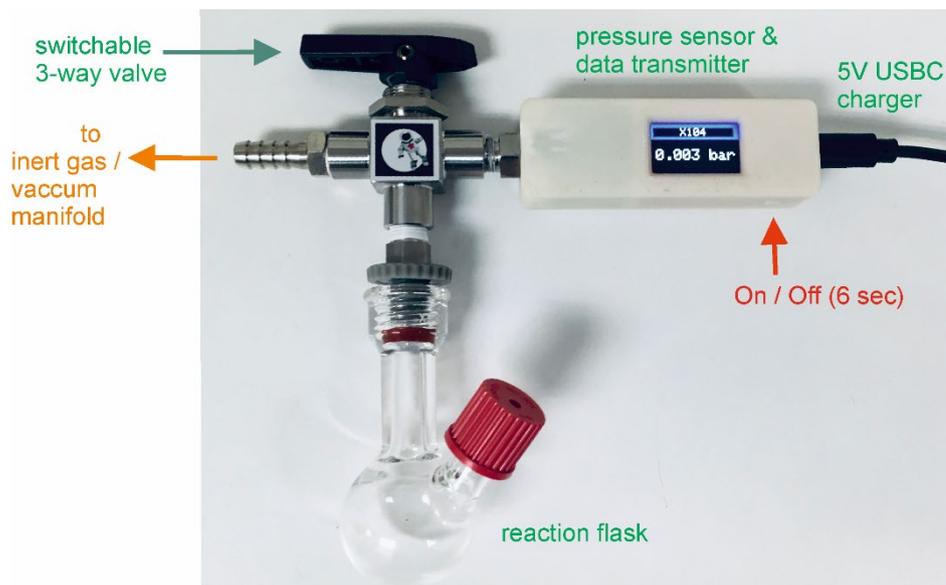
To activate the pressure sensor & data transmitter, either plug in the USBC charger or connect the internal battery by pressing the On/Off button. To power off the device, press and hold the On/Off button for 6 seconds. Please note that the internal battery has a maximum autonomy of approximately 40 minutes, which may not be sufficient for most experiments. It is recommended to keep the device plugged in throughout the duration of the experiments.

Running the X104v2BT application:

Open the X104_v2BT17 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.
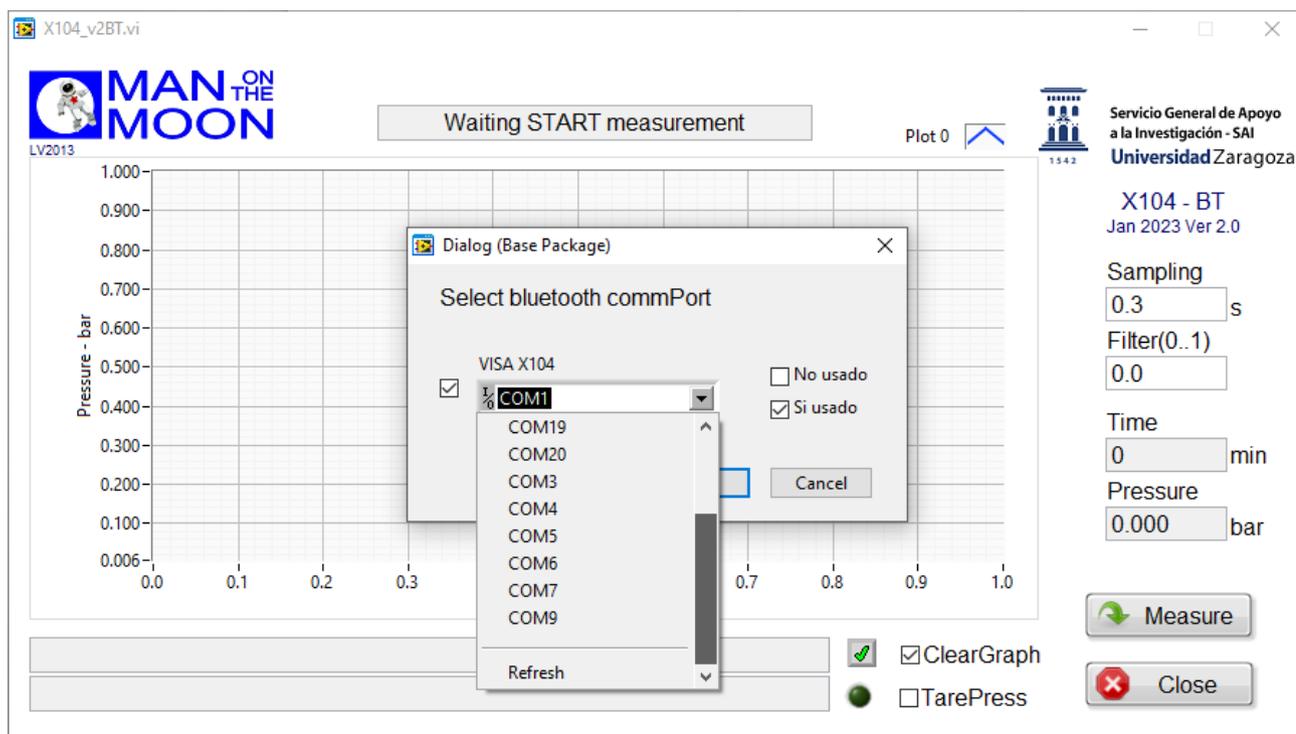
Clicking on the **Measure** button will prompt a dialog window to appear, allowing you to name and select the location for storing data. The data will be saved in a .dat ASCII file format, compatible with Excel and various other programs. Each **Sampling** interval (in seconds), the program writes the total reaction **Time** (in minutes) and the reaction **Pressure** (relative pressure in bar). The **Sampling** interval can be modified at any time by overwriting the previously entered value.

If necessary, a **Filter** function can be applied to reduce noise in the data. The formula is as follows:

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

A **Filter** value of 0 means no filter is applied.

The **ClearGraph** and **TarePress** options serve to reset the current **Time** and **Pressure** values to zero. These options do not erase the data already collected in the .dat file, nor do they alter the pressure displayed by the X104 pressure sensor.

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 **Close** exits the application.
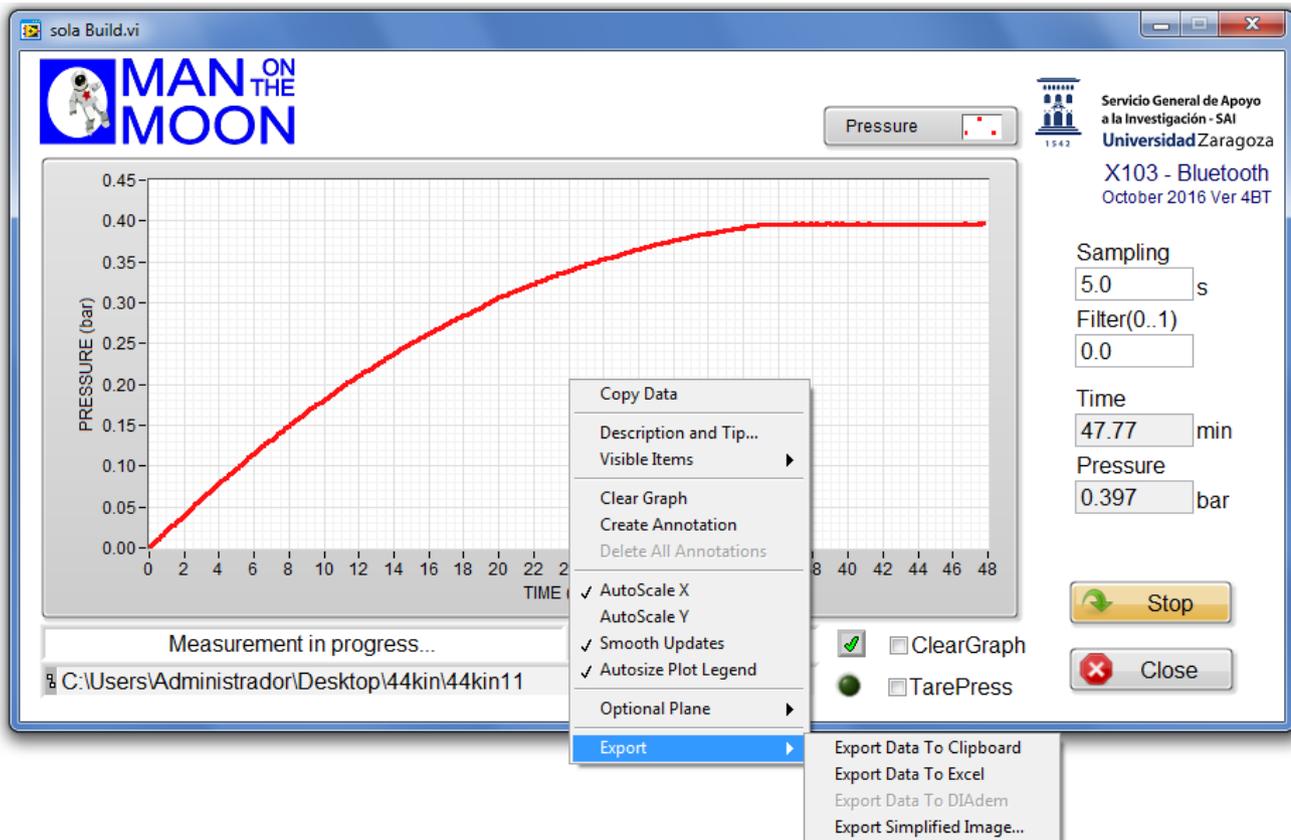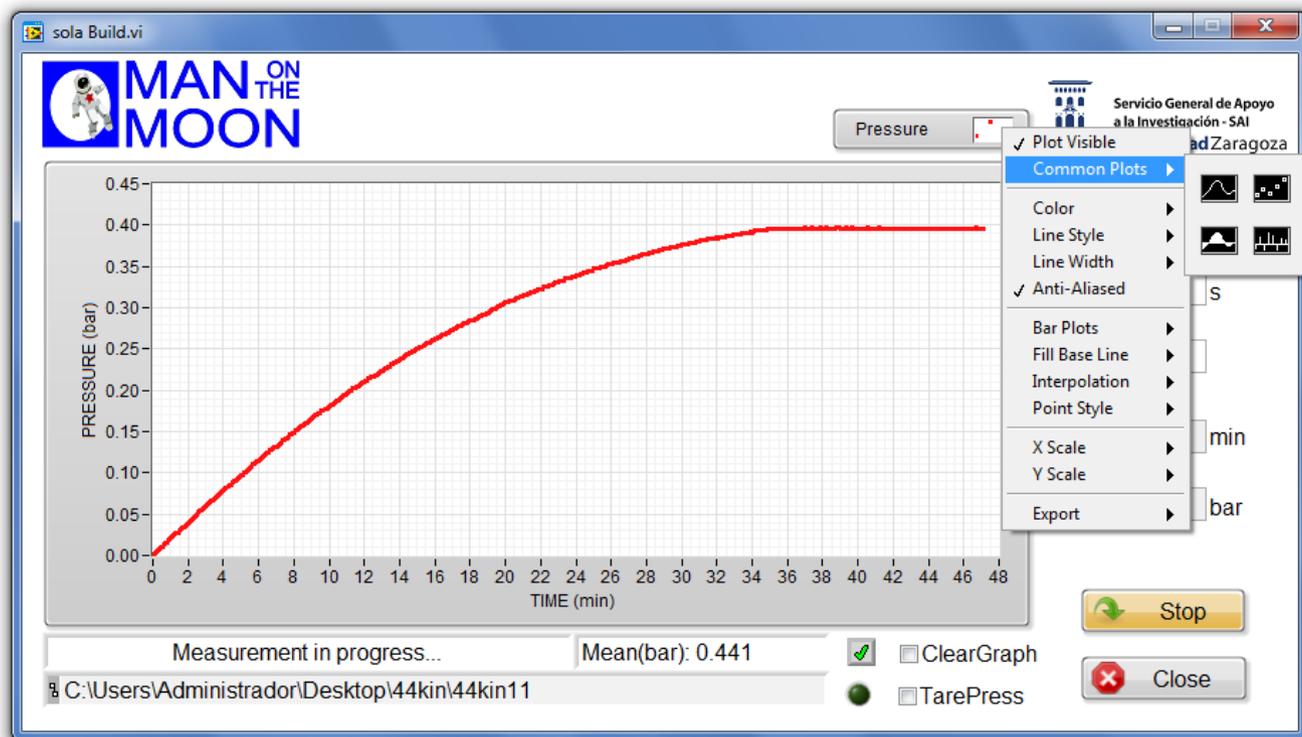
Comments on operation:

Gas evolution during the reactions is tracked through pressure increase. If necessary, users should conduct calibration of the reactor's total volume to convert pressure increase (in bar) into conversion (in mmol). As a reference, the preceding figures depict the evolution of a total of 0.30 mmol of O_2 from a reaction in 3 mL of solvent (water).

There exists an empty space between the 3-way valve and the pressure sensor, which may accumulate residual gases from previous operations. Prior to each experiment, it is advisable to clean this space to eliminate any lingering gases. Connect the system to a vacuum and toggle (180 degrees) the 3-way valve multiple times. Subsequently, repeat the same operation under inert gas. This procedure ensures that the aforementioned empty space is filled with inert gas, minimizing potential interference during experiments.

Reaction vessel options:

Each gas evolution kit includes a glass reactor, allowing customers to select the size that best suits their needs. The default option is the smallest reactor, with an approximate volume of 15 mL. Alternatively, customers may choose from larger options, with volumes of approximately 35 mL and 100 mL. For added convenience, additional or double-walled vessels, providing approximately 15 mL reaction volume and facilitating thermostatization, are available for separate purchase. Please feel free to contact us for pricing information and availability.

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