

### Bubbling System Description (Part# XET022):

The Bubbling System is designed to regulate and control the continuous flow of parahydrogen (or normal hydrogen for control experiments) gas through your sample inside a 5-mm NMR tube. The gas is bubbled through the sample and is then vented safely through the system and inside a fume hood (or other designated area) via pressure relief valves. The Mass Flow Controller (MFC) calibrated for hydrogen gas can be operated up to 125 psi (MAX) with a flow rate range of 0 to 150 sccm (standard cubic centimeters per minute). The MFC must not be exposed to over 150 psi pressure – otherwise permanent damage to the MFC may be caused. It is advisable to have a pressure regulator before MFC to ensure that maximum pressure is not exceeded.

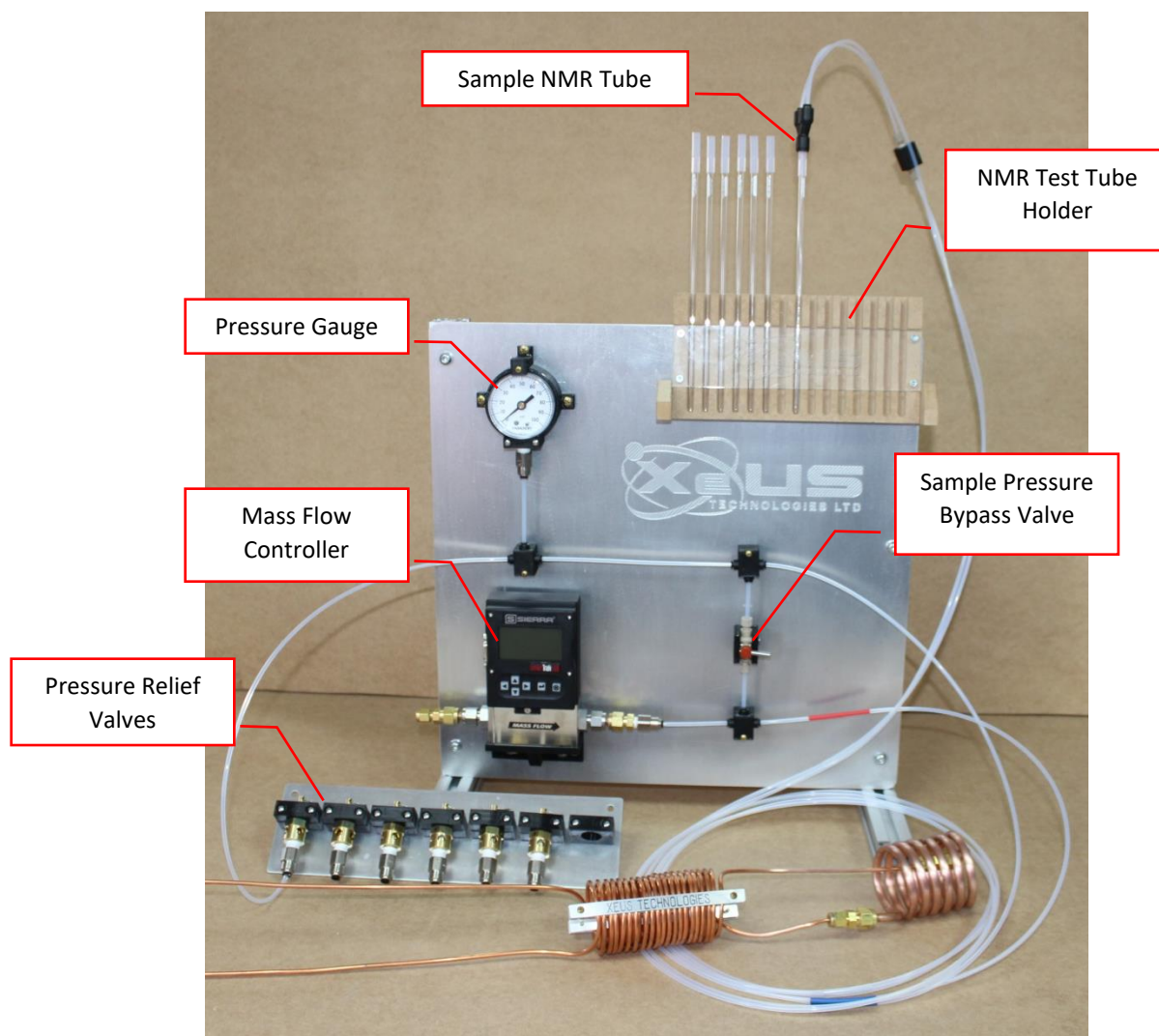


Figure 1: Bubbling System Setup indicating the various components of the system.

## Bubbling System Gas Flow Connectivity:

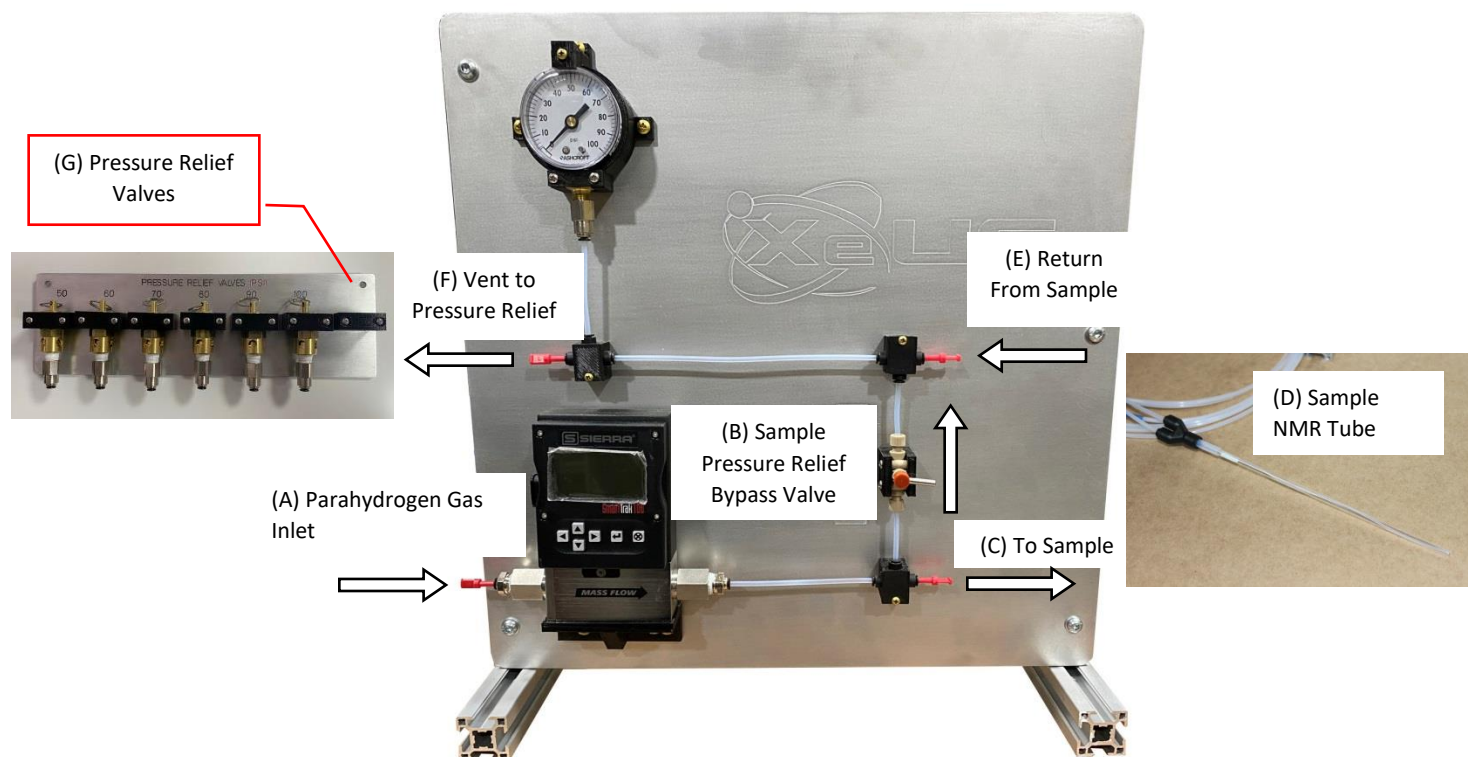


Figure 2: Bubbling System flow of gas through the system

## Bubbling System Set Up:

- Connect your parahydrogen gas line supply to the MFC Gas Inlet (A). The MFC comes equipped with a 1/8" push-to-connect adaptor. A 1/8" Compression fitting (<https://www.mcmaster.com/5272K291/>) is also supplied in the event you would like to make a more permanent connection (e.g., using copper lines). **Note:** In the event that you would like to replace the connector with another standard fitting or size, the MFC accepts 1/4" NPT Male thread.
- Connect the supplied 1/8" tubing the system vent (F) and the pressure relief valve (G) of the desired operating pressure. The Pressure Relief Valve plate consists of 6 relief valves set to different pressures: 50, 60, 70, 80, 90, and 100 PSI. The pressure relief valves maintain pressure inside the system while allowing the parahydrogen to vent. Do not operated more than 100 psi with a regular NMR tube.

**The Pressure Relief Valve Plate should be placed inside a working fume hood, to allow the gas to vent safely. Failure to do so while operating this system may result in a hazardous situation due to hydrogen gas being released inside an enclosed room.**



Figure 3: MFC gas inlet connector

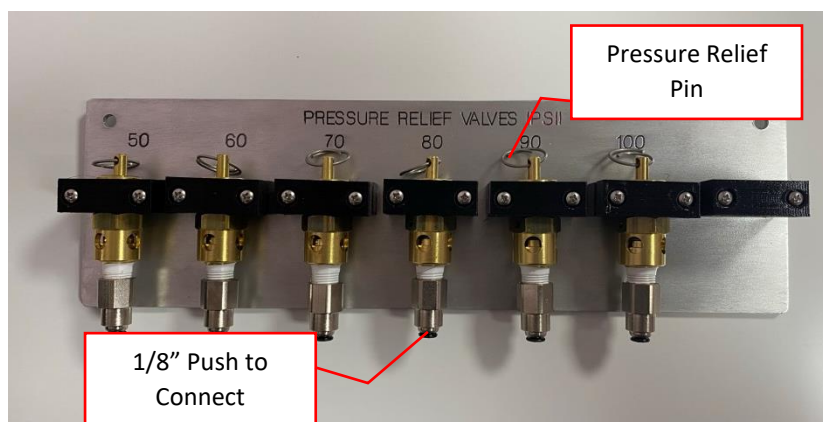


Figure 4: Pressure Relief Plate with 6 pressure relief valves ranging from 50 to 100 psi.

The pressure relief valves begin venting at ~3-5 psi before the rated cracking pressure. They are designed to not allow the system to exceed the rated pressure. If the lines are not pressurizing, then check to make sure the pressure relief pin (indicated in Fig. 4) of the valve is seated properly. The backing pressure of your parahydrogen should be at least a few psi higher than the set point of the relief valve to establish and maintain the flow of parahydrogen gas.

For instance: If you wish to operate the system at 70 psi, then connect the vent line from the bubbling system to the pressure relief valve set to 70 psi. Set your parahydrogen supply pressure to 80-125 psi. The system will then operate and be pressurized to 65-68 psi. The actual pressure can be monitored on the gauge (indicated in Fig. 1).

Once operating pressure is selected and the lines are connected, you may pressurize your lines. *Before you start pressurizing the system, make sure that the bypass valve is ON to make sure that the parahydrogen flow initially bypasses your sample to prevent vigorous bubbling through the NMR tube.*

- Next connect the supplied lines which connect to the NMR sample tube. The line with the red marking should be connected to port (C). This is the supply line of gas to the sample. The Return line is marked with blue and should be connected to port (D) (see picture below). 5-mm NMR tubed have been modified with 1/4" Teflon tubing to allow connection of the NMR tube to the 1/4" push-to connect Y connector.

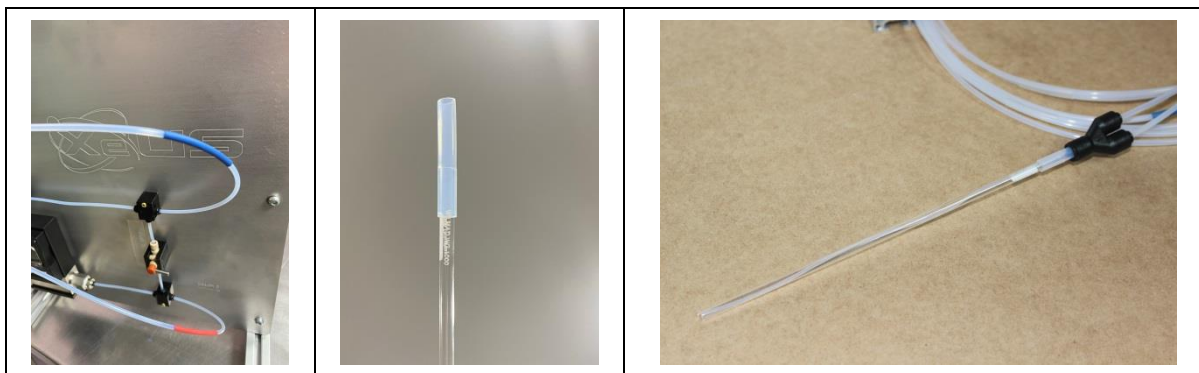


Figure 5: Gas line connectivity from the bubbling system to your NMR tube.

- Once all lines have been connected, you can then power on the MFC by connecting it to the supplied 24 VDC power supply with a DB15 connector. Please attach and secure the connector to the MFC as illustrated in the picture below.

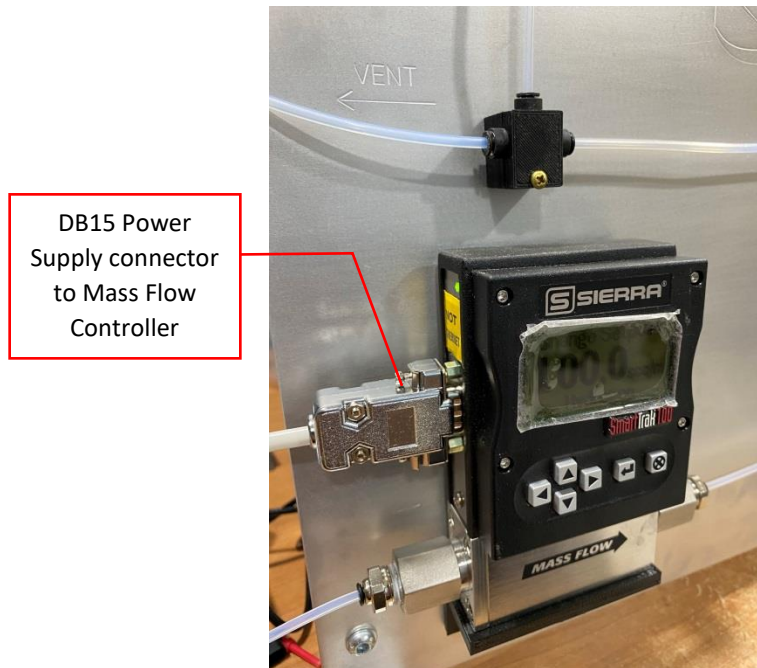


Figure 6: Mass Flow Controller Power Supply connection

- **The MFC Operating Parameters:**
  - Do not exceed maximum backing pressure of 150 psi, or this may result in damage to the controller.
  - The normal operating pressure range is up to 125 psi.
  - Flow Rate Range: 0 – 150 sccm

Please consult the supplied manufactures manual on operation of this device. A copy this manual can be found online at: <https://www.sierrainstruments.com/userfiles/file/manuals/100-series-instruction-manual.pdf>

### **Initial Recommended Operating Parameters:**

For the initial setting, please use 60 psi valve and set the flow rate to 50 sccm flow rate. These settings should allow the user to establish hydrogen bubbling through the sample. Once the bubbling has been established, the flow rate can be adjusted as needed.

### **Normal Operating Procedure:**

- 1) Connect all lines to the sample tube including the pressure relief valve as described above.
- 2) Have the Bypass Valve in the ON position (Fig2, B) to prevent vigorous bubbling;
- 3) Start the MFC by powering it ON;

- 4) Set the flow rate for example 50 sccm;
- 5) Watch for the overpressure to reach ~60 psi by monitoring the pressure gauge. To enable parahydrogen bubbling to your sample, turning OFF the bypass valve;
- 6) During the experiment, the bypass valve can be turned off/on at any given as needed;
- 7) When the experiment is over, and the system needs to be depressurized, turn the valve in the ON position to ensure the parahydrogen gas flow bypasses the NMR tube. Next, depressurize the system by depressurizing the pressure relief valve by pulling on the pressure relief pin. You can then safely remove your NMR tube from the Y Connector.

If you wish to keep the system depressurized while you connect your next sample and do not want to shut the system completely down, simply disconnect the vent line from the pressure relief valve, but keep the line in the fume hood for exhaust.

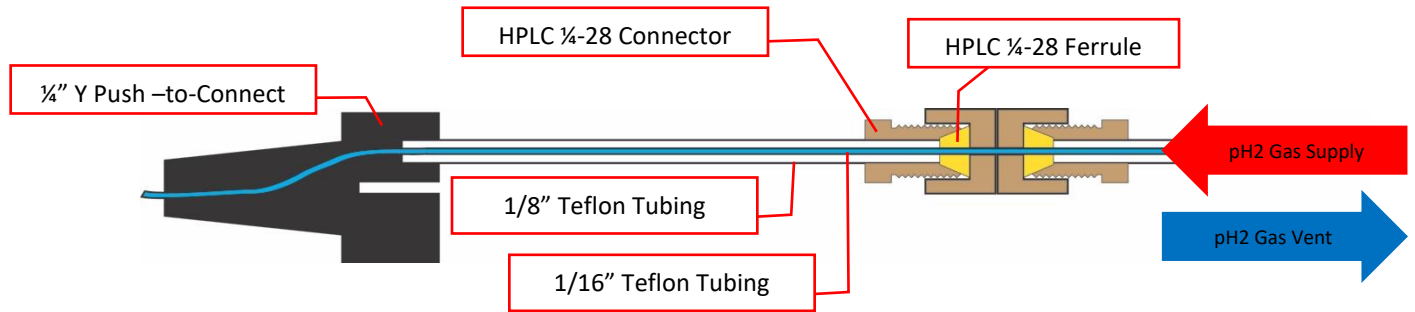
- If you desire to stop flow to the sample or change the valve (to higher or lower pressure), you may use the Sample Pressure Relief Bypass Valve (B). Opening this valve will allow the pressure of the system to be directed to the Vent, thus allowing the majority of the pressure to be diverted away from the sample.
- To turn off the system completely, simply turn off the power to the MFC, then followed by shutting of the parahydrogen gas supply. Make sure to depressurize the system by following Step 7.

### Replacing Consumables:

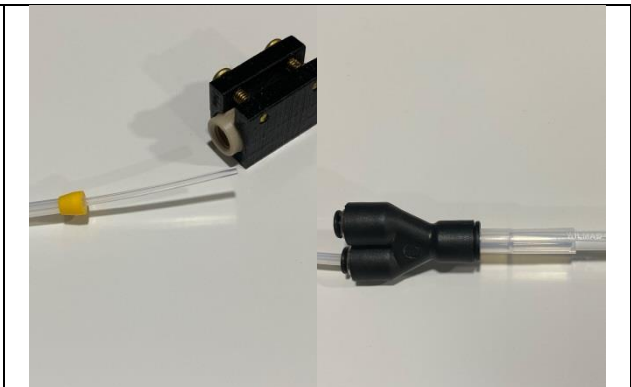

There are 4 consumables for the use of the system which you should have in stock:

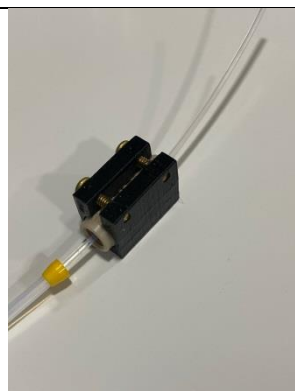
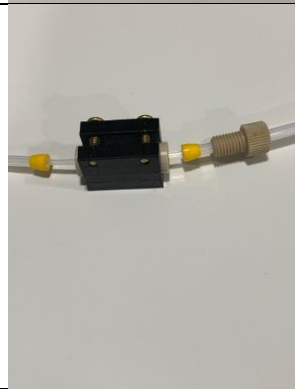


1. 1/16" Teflon tubing (For Specifications: <https://www.mcmaster.com/5239K23/>)
  2. 1/8" Teflon tubing (For Specifications: <https://www.mcmaster.com/5239K24/>)
  3. 1/4" Teflon tubing (For Specifications: <https://www.mcmaster.com/5239K12/>)
  4. 5 mm NMR Tubes (For Specifications: <https://www.wilmad-labglass.com/Products/WG-1000-8/>)
- **1/8" Tubing:** This is to replace any segments which may be accidentally damaged on the system.
  - **1/4" Teflon:** This is used to adapt 5mm NMR tubes to Y connector. If you would rather purchase ready modified NMR tubes, please contact us for assistance.
  - **5 mm NMR Tubes:** You will need replacement tubes to run your experiments.
  - **1/16" Teflon:** This size tubing is used to reach the bottom of your NMR tube, thus allowing the gas to bubble through your sample.

If you wish to purchase any of these supplies directly though XeUS Technologies. Please contact us.



This tubing will need to be cleaned regularly when using different samples, to prevent cross-contamination. Eventually, it may require a replacement. To replace this tubing, please follow the following procedure:

<p>First remove any NMR tube connected to the Y push-to-connect.</p> <p>Then remove the Y Connector from the 1/8" Teflon tubing, slowly guiding the 1/16" tubing through the connector until completely free.</p> <p>Proceed to unscrewing both HPLC 1/4-28 connectors. This will decompress the tubing, thus allowing you slowly pull the 1/16" Teflon tubing out of the 1/8" Tubing.</p>	
<p>Take new section of 1/16" Tubing and thread it through the 1/8" tubing which connects between the Y Connector and the compression fitting assembly.</p>	

<p>Thread the 1/16" tubing through the compression fitting assembly.</p>		
<p>Once through the compression assembly, feed the 1/16" tubing into the 1/8" supply line tubing as shown in the picture. Feed at least 50mm past the assembly into the tubing. Proceed to re-attaching the 1/4-28 HPLC connectors by for placing the 1/8" tube into the compression assembly, making sure the yellow ferrule is seated at on the end of the tube and inside the connector. Then slowly thread the HPLC connector until finger tight.</p>		
<p>Slowly feed the 1/16" tubing through the Y Connector making sure you leave a long enough segment to be able reach the bottom of your NMR tube. Then reconnect the 1/8" Tubing to the Y connector.</p>		
<p>To make sure you have a long enough segment of tubing, connect your NMR tube back on the Y connector. If your 1/16" tubing is too long, then cut the excess until the tubing sits as close to the bottom of your NMR tube.</p>	