

The MICRODOSER option of the GASPRO

INTRODUCTION

The MICRODOSER is an attachment to the GASPRO's high pressure sorption analyzer. This accessory was developed to enable the injection of very small gas doses on the tested material (down to the equivalent of 3 micromoles of hydrogen).

This feature is essential for characterizing small samples, typically less than 100 mg. It avoids total saturation of the sample's sorption capacity as soon as the first few doses are injected. In this way, the PCT curve obtained has a better resolution, and its analysis is therefore more reliable.

1. MICRODOSER's description

The MICRODOSER option is a complete system with its own:

- calibrated volume of about 0.5 ml, which is integrated in the MICRODOSER's enclosure,
- pressure sensor which, when the MICRODOSER is in operation, is used in place of the one integrated in the GASPRO,
- sample holder, connected to the MICRODOSER's outlet by a 1/16" tube for easy handling and insertion in a standard furnace.

All are connected by a semi-automatic 4-way pneumatic valve. The valve has four positions allowing, in a sequential way: to place the sample at the initial pressure/vacuum level, to load the calibrated volume with a dose of test gas, or to inject this dose into the sample holder.

This technology is patented under the number US8132476.

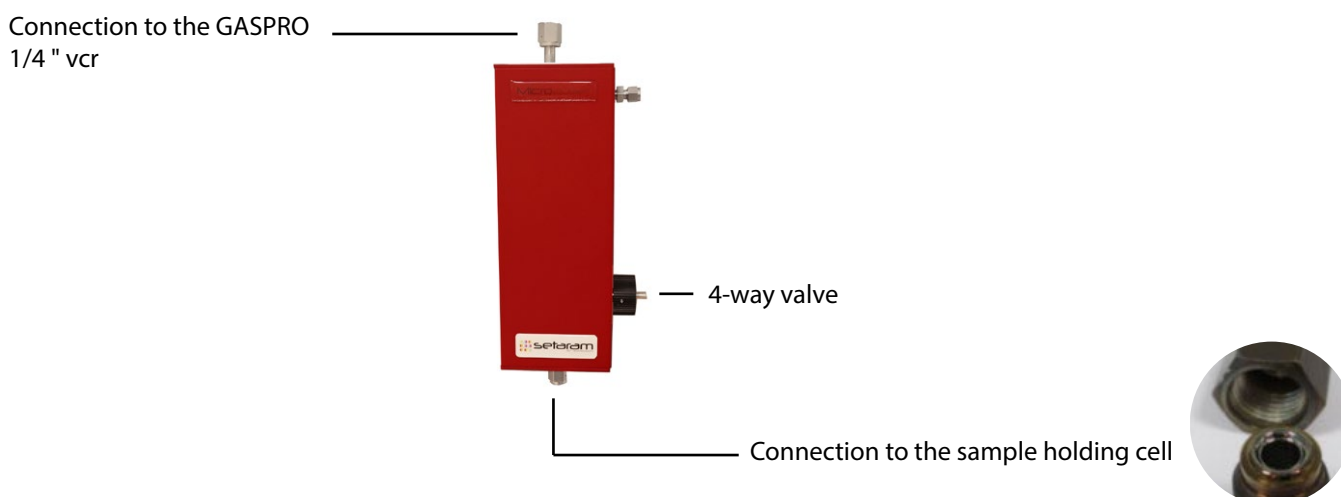


Figure 1 – MICRODOSER's attachment

Maximum sample volume	0.5 ml
Maximum pressure	60 bar
Maximum temperature	400 °C

2. MICRODOSER's operation

To operate, the MICRODOSER is first connected tightly to the outlet of the GASPRO as any sample holder would be. The electrical connections of the pressure sensor and of the thermocouple of the MICRODOSER are made, as well as the pneumatic connections of the valve.

Then, an experiment takes place in 3 steps:

- loading the sample into the sample holder. The sample holder is then tightly connected to the MICRODOSER with the suitable seals.
- placement of the oven (if used) and the thermocouples on the sample holder.
- Configuration of the software and setup of the experiment, which may include, as with many GASPRO tests: dead volume calibration, purging, sample degassing, sample loading, and PCT, kinetic, or cycle-life testing procedures.

3. Test example

A 5.26 mg sample of palladium was tested using GASPRO and the MICRODOSER attachment using the PCT mode. This mode allows the injection of gas doses of increasing pressure on a sample and to measure the quantities of adsorbed gas at the end of each injection.

In the present case, the gas was hydrogen, and the maximum pressure was set to 8 bar. The sample was heated to 170°C. Gas doses were prepared in the MICRODOSER's reservoir volume (0.489 ml), with a 0.5 bar delta P, corresponding to about 10 micromoles of hydrogen.

Figure 2 shows the PCT diagram of this experiment, showing typical plateau at 2.85 bar observed for palladium at that temperature. The total hydrogen quantity adsorbed by the sample was equivalent to 16 micromoles of hydrogen.

This example illustrates the capacity of the MICRODOSER attachment to inject very small doses of gas on samples of a few milligrams and accurately characterize their sorption properties. On top of such PCT curves, Kinetics and Cycle-Life measurements can be also achieved using the MICRODOSER attachment.

For more examples check technical note TN629, which shows small samples characterization with sample weights in the 20-50 mg range.

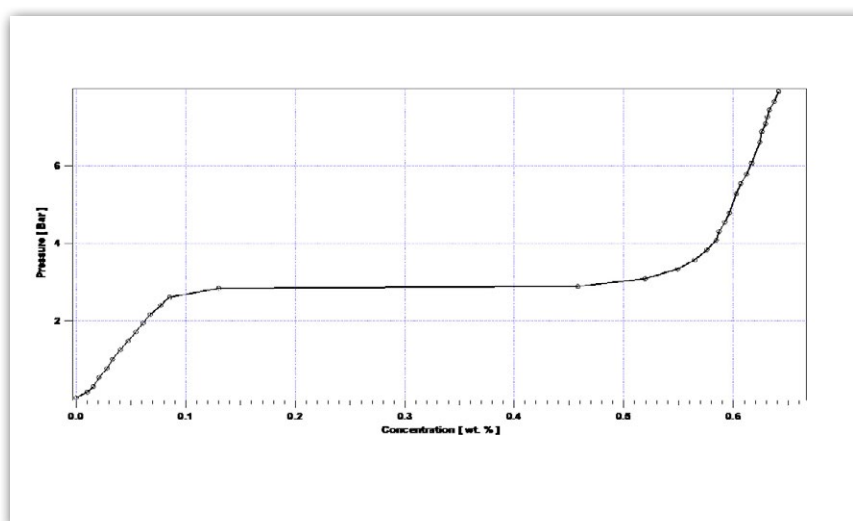


Figure 2 – Pressure-Composition-Temperature diagram of a 5.26 mg palladium sample at 170 °C