

The CRYOPRO option of the GASPRO

INTRODUCTION

The CRYOPRO is an attachment to the GASPRO's high pressure sorption analyzer. This accessory was developed to enable gas sorption measurements at extremely low temperatures (down to -260°C). Low temperature operation is critical to characterize porous materials that interact with gases through physisorption. This is especially important when these materials are considered for gas storage or separation applications.

EXPERIMENT

1. CRYOPRO's description

The CRYOPRO consists mostly of a cryostat, a sample holder and two external temperature controllers.

- The cryostat is shown in Figure 1. It is connected to a 100 liters liquid nitrogen or liquid helium tank to provide the low temperatures. The chosen cryogenic liquid flows into the bottom of the cryostat's Dewar where it is evaporated by a small heating element.
- A pressure resistant sample holder (Figure 2) is inserted into the cryostat. It is equipped with a manual valve (for airless sample transfer), a temperature probe (fixed thermocouple or Pt temperature probe) and a furnace that can be directly placed around the sample holder. For small samples or steady operations at liquid nitrogen or liquid helium's temperatures the evaporative temperature controller is sufficient for maintaining a steady temperature. To measure larger samples at temperatures above the cryogenic liquid temperatures, the sample holder's furnace is required.
- The cryostat's temperature is kept constant thanks to its own temperature controller, and when used, the sample holder's furnace and thermocouples are connected to another controller. Ensuring a stable temperature for experiments at low temperatures is a crucial factor for high accuracy measurement.

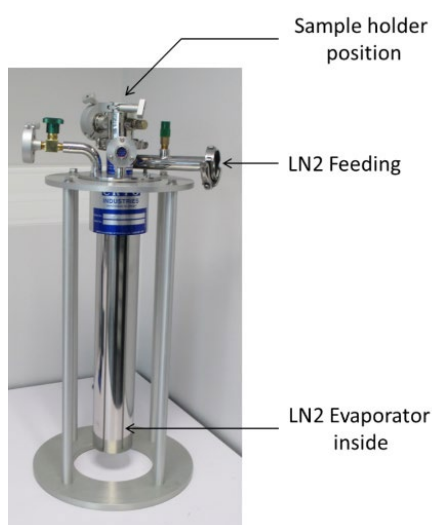


Figure 1 – CRYOPRO's cryostat

Using liquid nitrogen in the 100 liters tank provides an autonomy of up to 50h to the system.

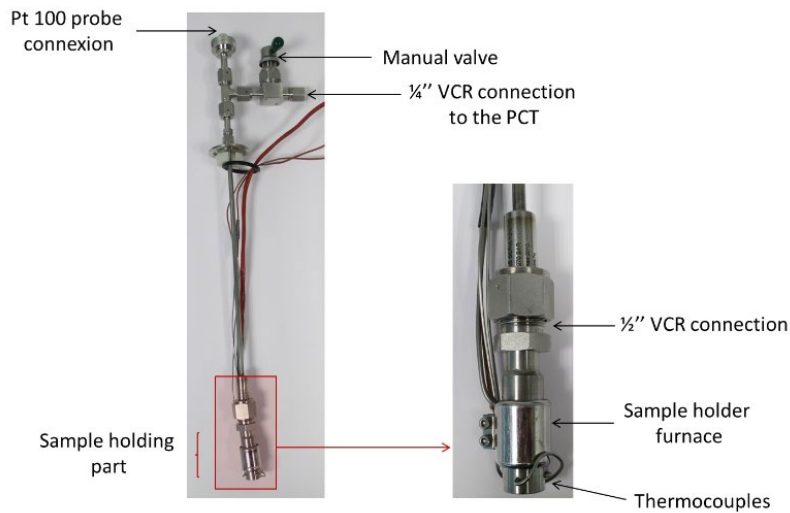


Figure 2 – CRYOPRO's sample holder

Maximum sample volume	3 ml
Maximum pressure	200 bar
Maximum temperature	200 °C when outside the cryostat
	100 °C when inside the cryostat

2. CRYOPRO's operation

Using the CRYOPRO attachment goes through 5 main steps:

- Sample loading, which can be done in the lab atmosphere or in a glovebox if the sample is air-sensitive. The sample holder is then tightly closed using the adapted seals.
- Placing the furnace (if used) and thermocouples on the sample holder, inserting the sample holder in the cryostat, and attaching the sample holder to the GASPRO.
- Electrical connections of the heaters and thermocouples to the controllers and of the controllers to the GASPRO.
- Setting up the flow of cryogenic liquid in the cryostat to reach the desired start temperature.
- Software configuration and experiment setup, that may include as many GASPRO tests: dead volume calibration, purge, sample degassing, sample charging, and PCT, kinetics, or cycle-life testing procedures.

3. Test example

A carbon sample was tested using GASPRO and the CRYOPRO attachment with 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 70 bar. Results obtained at two temperatures are overlaid on Figure 3 below:

- at 298 K without using the cryostat,
- at 77 K with the sample holder inserted in the cryostat and liquid nitrogen flowing in the cryostat.

On top of such PCT curves, Kinetics and Cycle-Life measurements at low temperatures can be also achieved using the CRYOPRO attachment.

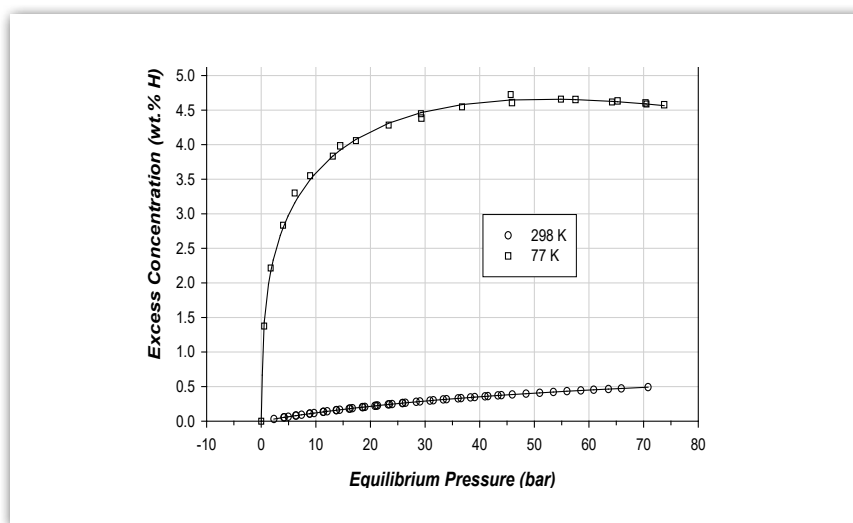


Figure 3 – Pressure-Composition-Temperature diagram of a carbon sample at 77 K and 298 K