

## MS17-P7 A controlled pressure/temperature set-up for synchrotron in situ studies of solid-gas processes and reactions: Case of the structural deformation of ZIF-8

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A novel set-up has been designed and used for synchrotron radiation X-ray high-resolution powder diffraction (SR-HRPD) in transmission geometry for in situ solid-gas reactions and processes in an isobaric and isothermal environment. The pressure and temperature of the sample are controlled from 10<sup>-3</sup> to 1000 mbar and from 80 to 1000 K, respectively. To test the capacities of this novel experimental set-up, structure deformation in the porous material zeolitic imidazole framework (ZIF-8) by gas adsorption at cryogenic temperature. The adsorption properties of ZIF-8 for a variety of strategic gases, shows unusual multi-stepped adsorption features for the adsorption of various gas probes (i.e., N<sub>2</sub>, CO, Ar, O<sub>2</sub>) [1-3]. There seems yet to be a dearth in the understanding of the gas adsorption properties of flexible materials [3,4]. X-ray diffraction experiments were conducted at the Spanish CRG BM25 SpLine at ESRF in a controlled environment chamber with fine control of the gas dosage, sample outgassing under vacuum, temperature control and simultaneous SR-HRPD recording [4]. Figure 1 shows the nitrogen adsorption/desorption isotherm of ZIF-8 at 85K and the SR-HRPD diffractograms (inset) corresponding to various gas loadings. Measurements were fast enough to observe the evolution of the crystallographic phases of the material, making possible to determine structural changes in the solid at in operando conditions [4].

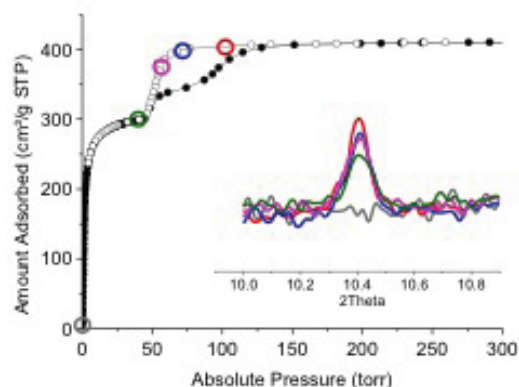
The real time monitoring of the SR-HRPD patterns of ZIF-8 indicate that the gas-induced structural flexibility is linked to the organization of the adsorbed gas molecules in the adsorption sites, as well as the polarizability and molecular size and shape of the gases.

### REFERENCES

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**Figure 1.** Equilibrium N<sub>2</sub> adsorption/desorption isotherm 85 K of ZIF-8 and (inset) real time SR-HRPD during gas release (desorption branch).

**Keywords:** XRPD; isobaric/isothermal environment, ZIF-8, adsorption, solid-gas reactions