

Supporting Information

An integrated stopped-flow device for the study of porous materials using hyperpolarized ^{129}Xe NMR

Jing Li,[‡] Estelle Léonce,[‡] Corentin Coutellier,[‡] Céline Boutin,[‡] Kévin Chighine,[‡] Charles Rivron,[‡]

Anne Davidson,[§] and Patrick Berthault^{‡}*

[‡] NIMBE, CEA, CNRS, Université de Paris Saclay, CEA Saclay, 91191 Gif-sur-Yvette, France

[§] Laboratoire de Réactivité de Surface, Sorbonne Universités, UPMC Université Paris 06, UMR CNRS 7197, 4 Place Jussieu, 75005 Paris, France

- **Figure S1.** Front and back of the used data acquisition board for dual pressure measurement.
- **Figure S2.** USB to UART RS485 protocol bridge module front and back.
- **Figure S3.** N_2 adsorption and desorption isotherms of MCM-41.
- **Table S1.** Pore volume, pore size and surface area of MCM-41 as derived from N_2 sorption.
- **Figure S4.** Continuous hyperpolarized ^{129}Xe spectra of MCM-41 after elimination of the organic template in pores by calcination under 900°C for 8 hours.
- **Figure S5.** Self-diffusion data points for peak F recorded in the first two gradient loops.
- **Figure S6.** TOPSPIN pulse program for the used HP ^{129}Xe self-diffusion experiments.
- **Figure S7.** TOPSPIN pulse program for the used HP ^{129}Xe EXSY experiments.

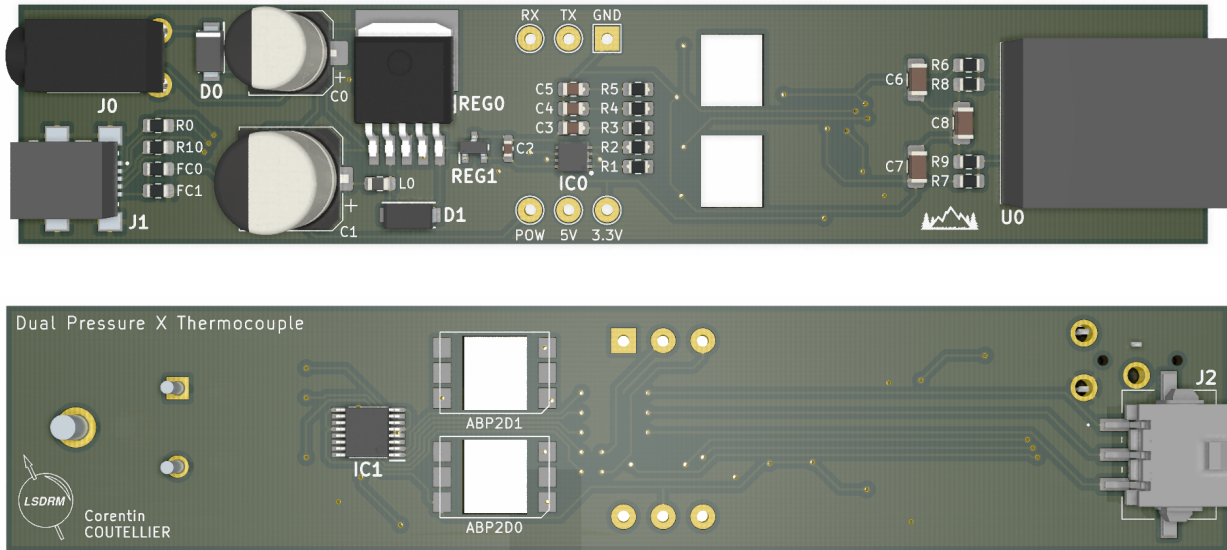


Figure S1. Front and back of the used data acquisition board for dual pressure measurement. Empty spaces in ABP2Dx areas are for soldering the pressure sensors (ABP2MANT010BAAA5XX).

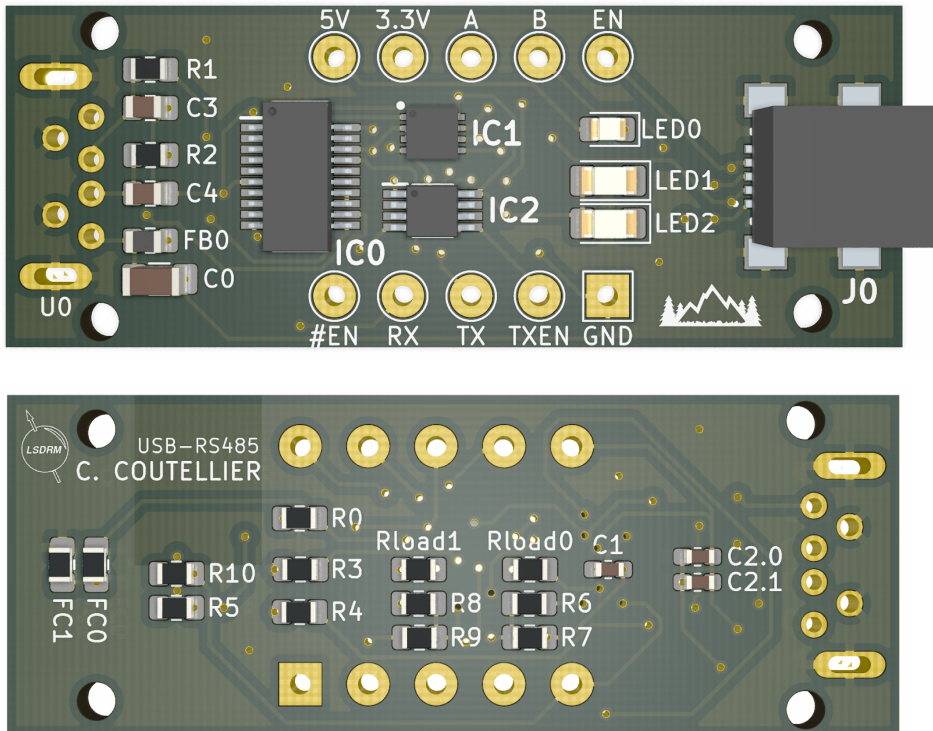


Figure S2. USB to UART RS485 protocol bridge module front and back. At the left of the front part, a USB connector (MC32603) is soldered to connect the module to a computer.

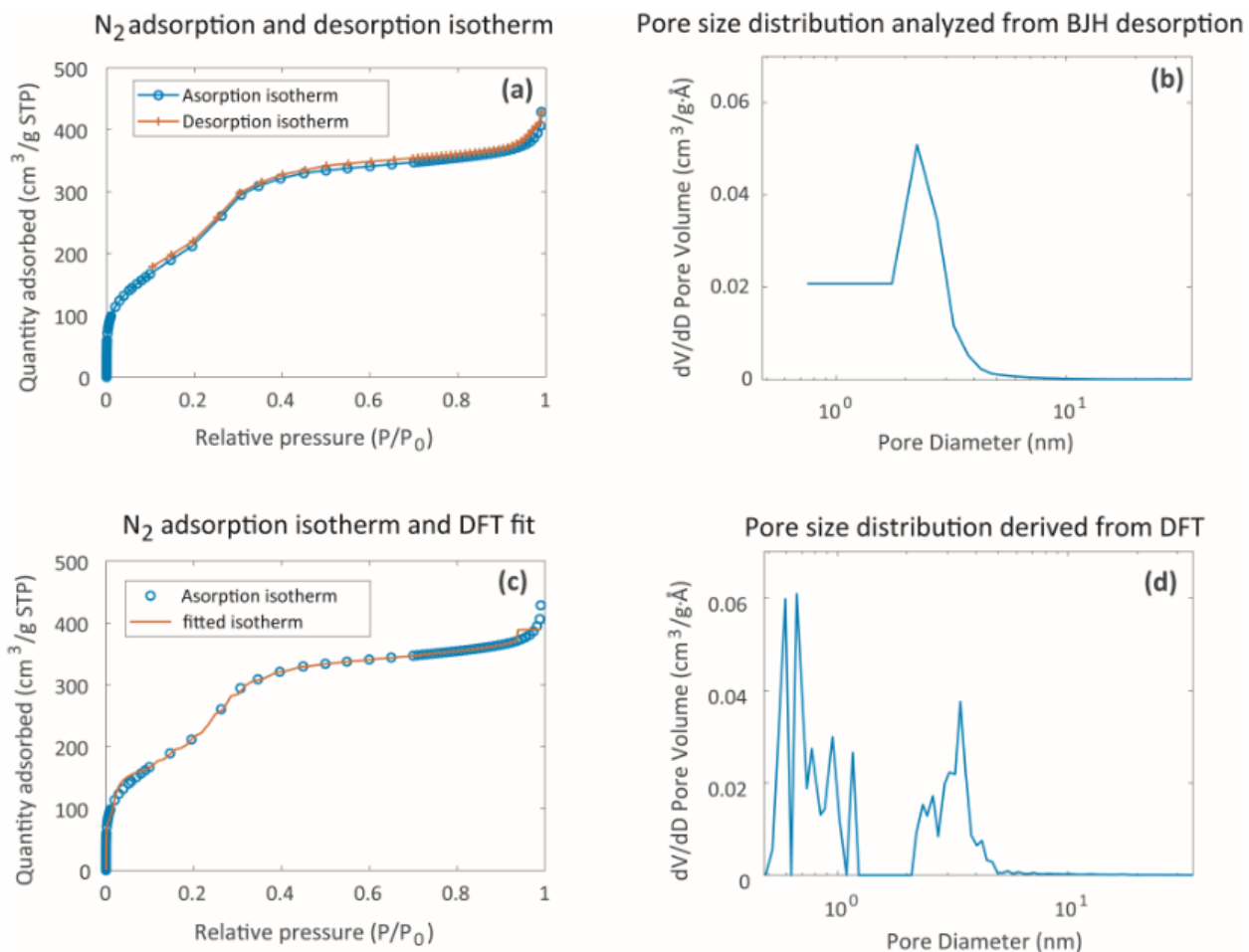


Figure S3. (a) N₂ adsorption and desorption isotherms of MCM-41; (b) pore size distribution derived from the BJH desorption analysis; (c) NLDFT fit of the adsorption isotherm and (d) pore size distribution derived from NLDFT analysis.

BET surface area (m ² /g)	DFT surface area (m ² /g)	BJH pore volume (cm ³ /g)	DFT pore volume (cm ³ /g)
885.8017	1307.951	0.74	0.60

Table S1. Pore volume, pore size and surface area of MCM-41 as derived from N₂ sorption.

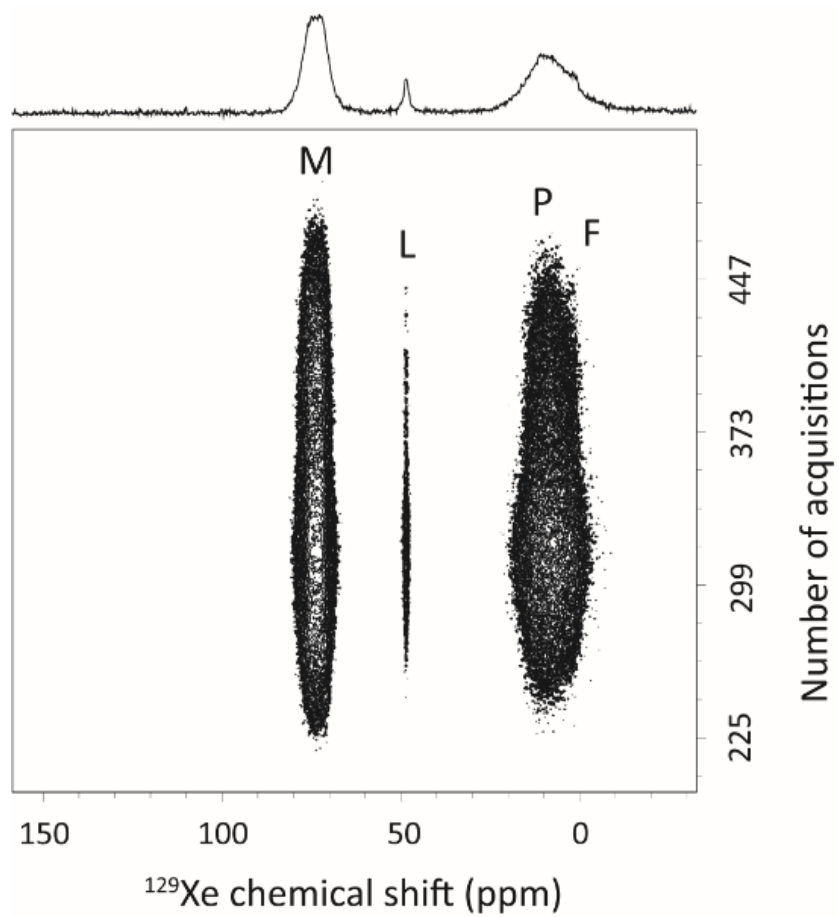


Figure S4. Continuous hyperpolarized ^{129}Xe spectra of MCM-41 after elimination of the organic template in pores by calcination under 900°C for 8 hours.

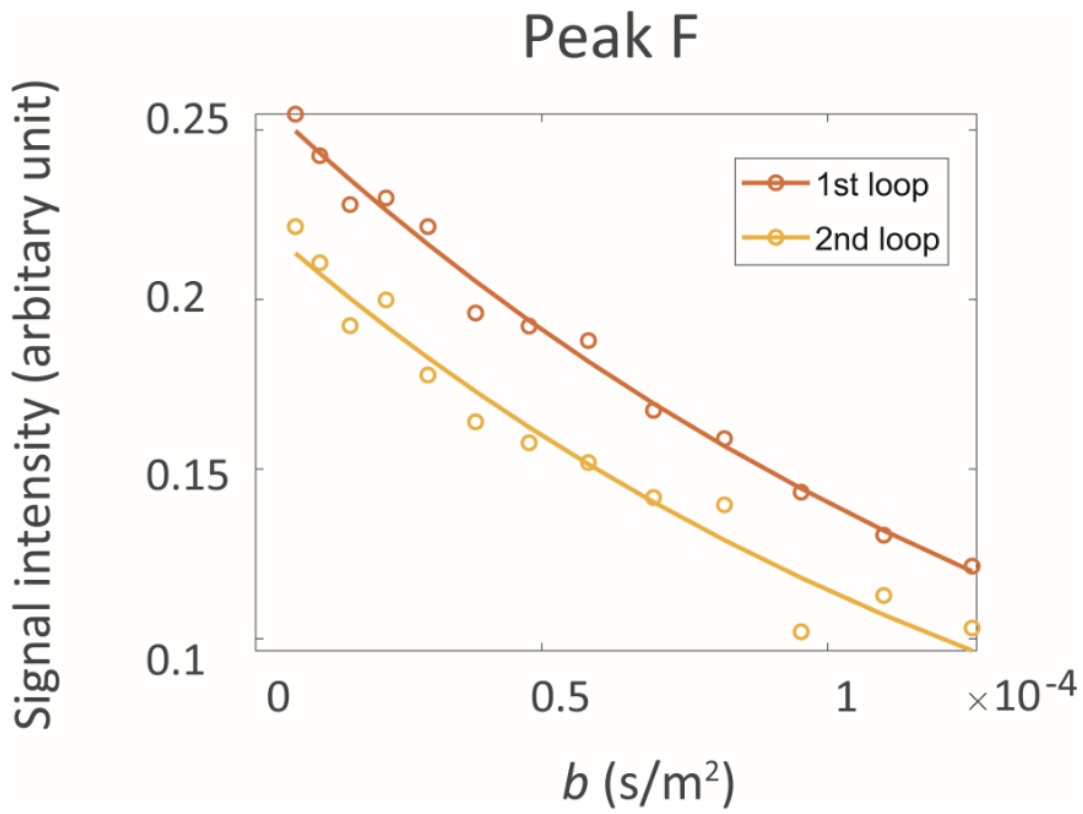


Figure S5. Self-diffusion data points for peak F recorded in the first two gradient loops. Extracted self-diffusion D values: $6.2 \cdot 10^{-6} \text{ m}^2/\text{s}$ for the first loop; $6.7 \cdot 10^{-6} \text{ m}^2/\text{s}$ for the second loop.

;SELF-DIFFUSION EXPERIMENT (PB)

;avance-version

;from stegp1s

;2D sequence for diffusion measurement using stimulated echo using 1 spoil gradient

#include <Avance.incl>

#include <Grad.incl>

#include <Delay.incl>

define list<gradient> diff=<Difframp>

*"DELTA1=d20-p1*2-p30-d16-p19-d16"*

"l2=td1/l1"

1 ze

30m

2 1m setnmr3|28

d10

20u setnmr3^28

4 d1

p0 ph4

go=4 ph30

d11 wr #1 if #1 zd

; Start of DOSY sequence

5 1m setnmr3|28

d10 UNBLKGRAMP

20u setnmr3^28

d1

p1 ph1

*p30:gp6*diff*

d16

p1 ph2

d12

p19:gp7

d16

DELTA1

p1 ph3

*p30:gp6*diff*

d16

4u BLKGRAMP

go=5 ph31

d11 wr #0 if #0 zd

5m igrad diff

lo to 2 times l1

; number of points in the indirect dimension of each DOSY

5m zgrad diff

lo to 2 times l2

; number of DOSY experiments

exit

ph1= 0 0 0 0 2 2 2 2 1 1 1 1 3 3 3 3

ph2= 0 2

ph3= 1 3 0 2

ph4=0 2

ph30=0 2

ph31=0 0 2 2 2 2 0 0 3 3 1 1 1 1 3 3

```

; d16: delay for gradient recovery
; d20: diffusion time (big DELTA)
; td1: number of experiments
; FnMODE: QF
; use xf2 and DOSY processing
; use gradient ratio: gp 6 : gp 7 100 : -17.13 or else :-)

```

Figure S6. TOPSPIN pulse program for the used ^{129}Xe self-diffusion experiments.

```

;EXSY experiment (PB)
;avance-version
;from noesyph

#include <Avance.incl>

"in0=inf1 "

1 ze
  30m
2 d1 setnmr3 | 28 ; Activate syringe pump and open gas
circuit
  d10 ; push duration
  20u setnmr3^28 ; Stop syringe pump and close circuit
4 d1
  p1 ph1
  d0
  p1 ph2
  d8
  p1 ph3
  go=2 ph31
  d11 mc #0 to 2 F1PH(calph(ph1, +90), caldel(d0, +in0))
exit

ph1=(360) 45 225
ph2=0 0 0 0 0 0 0 0 2 2 2 2 2 2 2 2
ph3=0 0 2 2 1 1 3 3
ph31=0 2 2 0 1 3 3 1 2 0 0 2 3 1 1 3

```

Figure S7. TOPSPIN pulse program for the used ^{129}Xe EXSY experiments.