Electronic Supplementary Material (ESI) for Dalton Transactions.

---Supporting Information---

A combination of Proton Spin Diffusion NMR and molecular simulations to probe supramolecular assemblies of organic molecules in nanoporous materials

Eddy Dib,[a] Beatriz Bernardo-Maestro,[b] Fernando López-Arbeloa,[c] Joaquín Pérez-Pariente,[b] and Luis Gómez-Hortigüela[b]**

[a] Normandie Univ, ENSICAEN, UNICAEN, CNRS, Laboratoire Catalyse et Spectrochimie, 14000 Caen, France
[b] Instituto de Catálisis y Petroleoquímica-CSIC, C/Marie Curie 2, 28049, Cantoblanco, Madrid, Spain
[c] Departamento de Química Física, Universidad del País Vasco, Apartado 644, 48080, Bilbao, Spain
E-mail : eddy.dib@ensicaen.fr, lhortiguela@icp.csic.es

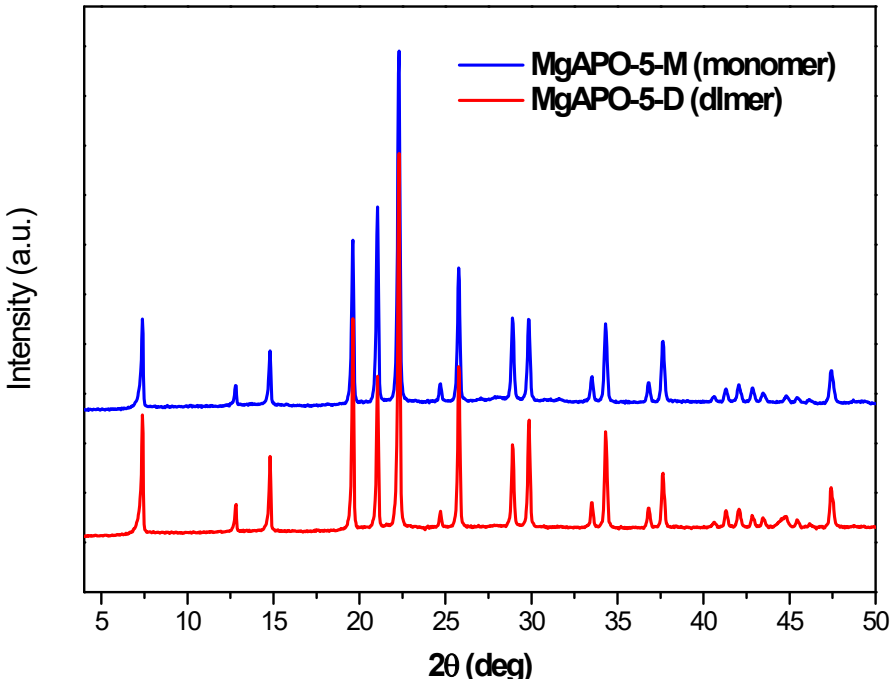

Figure S1. XRD patterns of MgAPO-5-M (with EPH monomers) and MgAPO-5-D (with EPH dimers) samples.

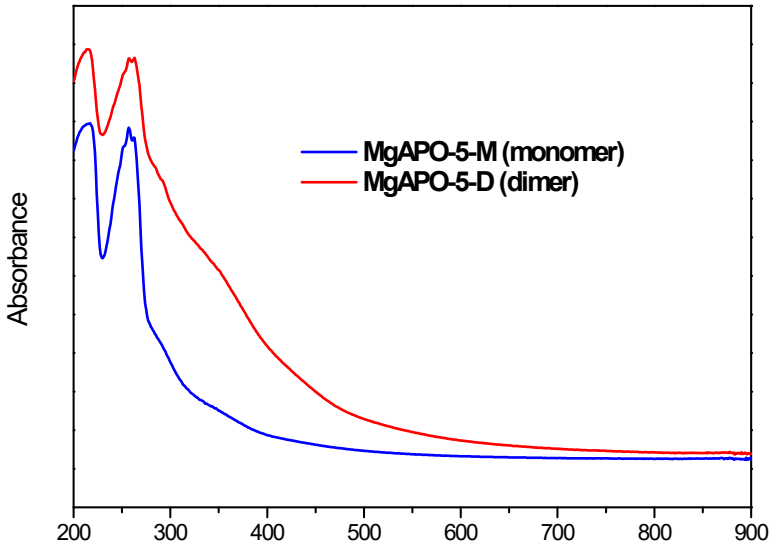

Figure S2. Diffuse Reflectance UV-Vis spectra of MgAPO-5-M (a) and MgAPO-5-D (b).

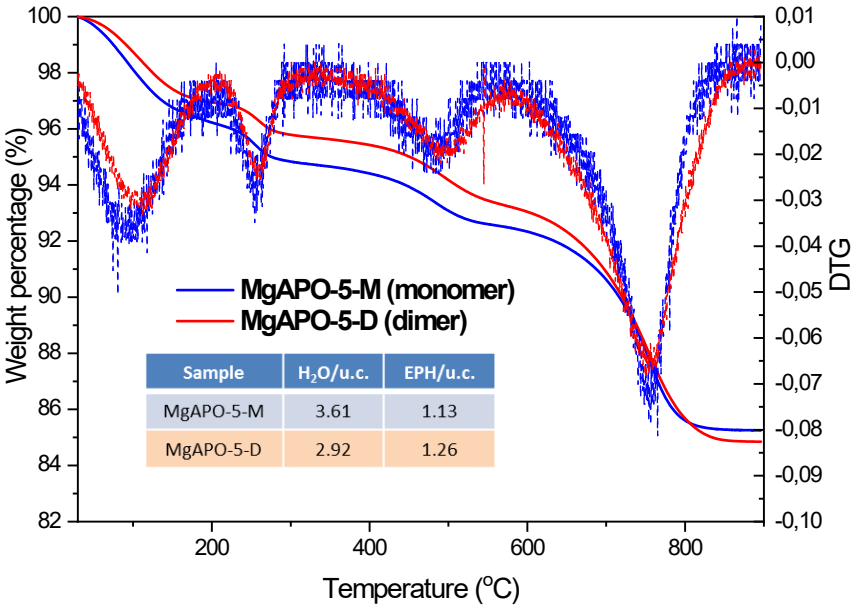

Figure S3. TGA of MgAPO-5-M (blue line) and MgAPO-5-D (red line), and their corresponding water and EPH contents per unit cell.

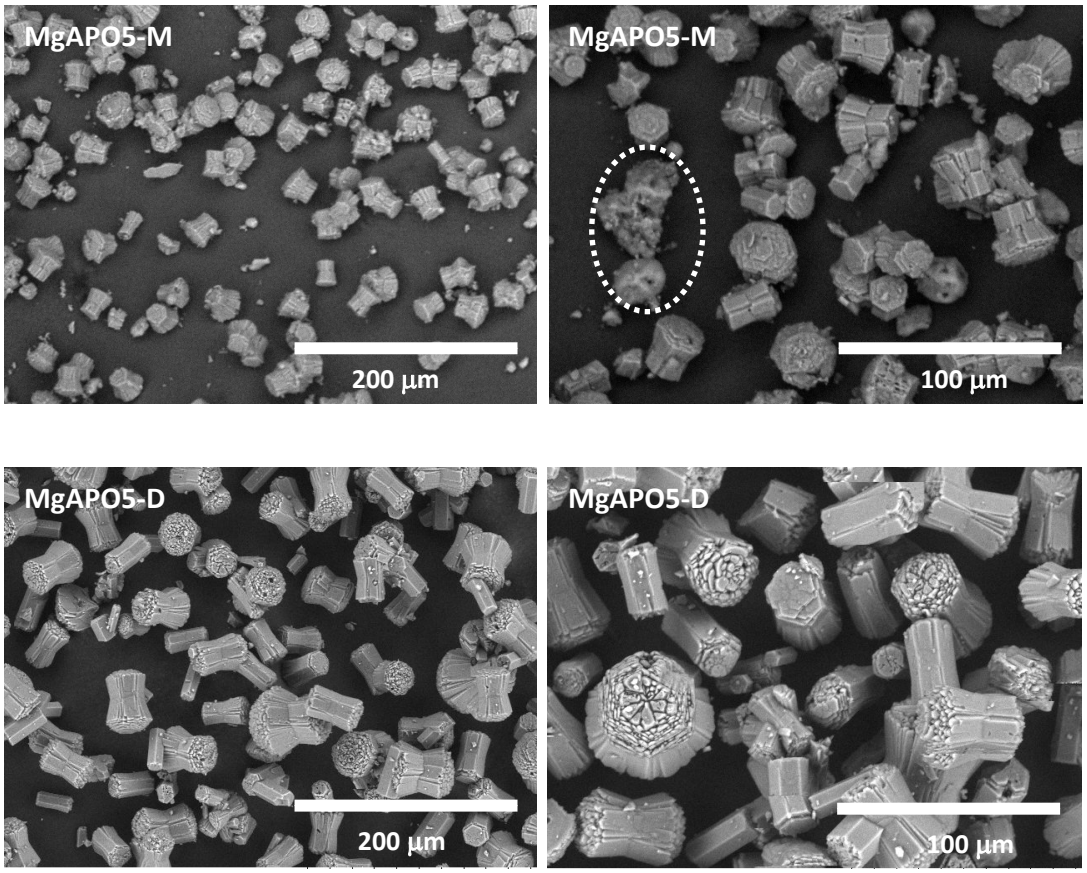

Figure S4. SEM pictures of MgAPO5-M (with monomers, top) and MgAPO5-D (with dimers, bottom) samples, with x500 (left) and x1000 (right) magnifications. Dashed circle shows the presence of some amorphous material.

Solid State MAS-NMR spectra of the solid samples were recorded with a Bruker AV 400 WB spectrometer, using a BL7 probe for ^{13}C and a BL4 probe for ^{31}P and ^{27}Al. ^{1}H to ^{13}C Cross-Polarization spectra were recorded using π/2 rad pulses of 2.75 μs for ^{1}H, a contact time of 3 ms and a recycle delay of 4 s. For ^{31}P, π/2 rad pulses of 4.25 μs and recycle delays of 80 s were used. For ^{27}Al, π/12 rad pulses of 1 μs and recycle delays of 1 s were used. All these spectra were recorded while spinning the samples at *ca.* 11.2 kHz.

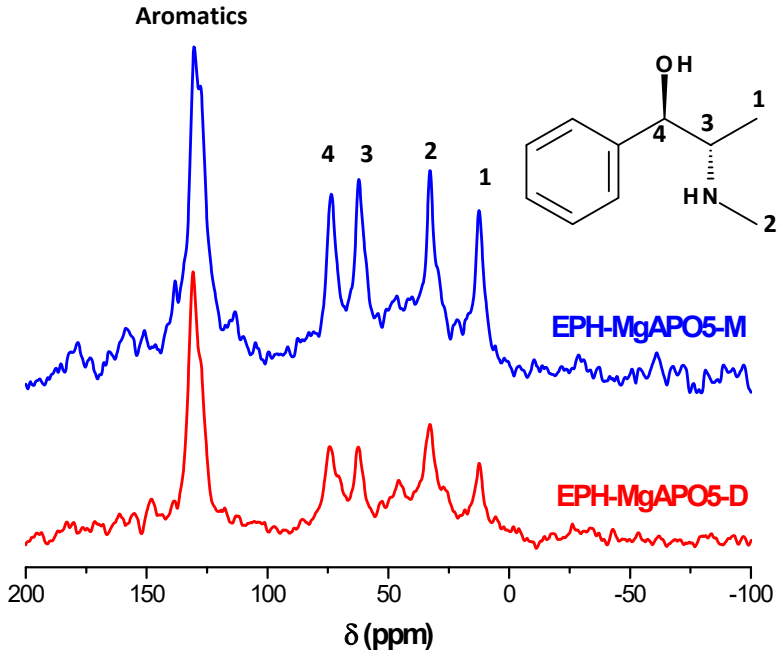

Figure S5. ^{13}C NMR spectra of MgAPO-5-M (top, blue line) and MgAPO-5-D (bottom, red line).

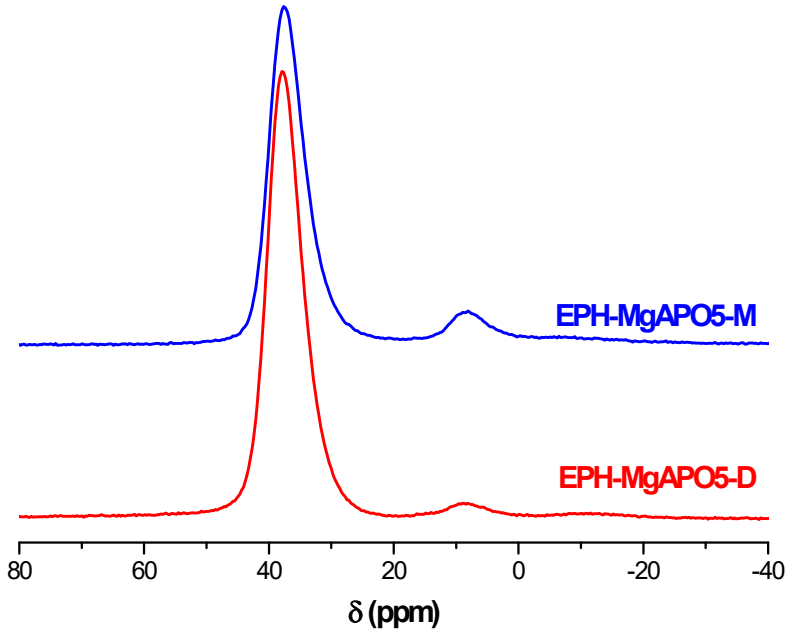

Figure S6. ^{27}Al NMR spectra of MgAPO-5-M (top, blue line) and MgAPO-5-D (bottom, red line).

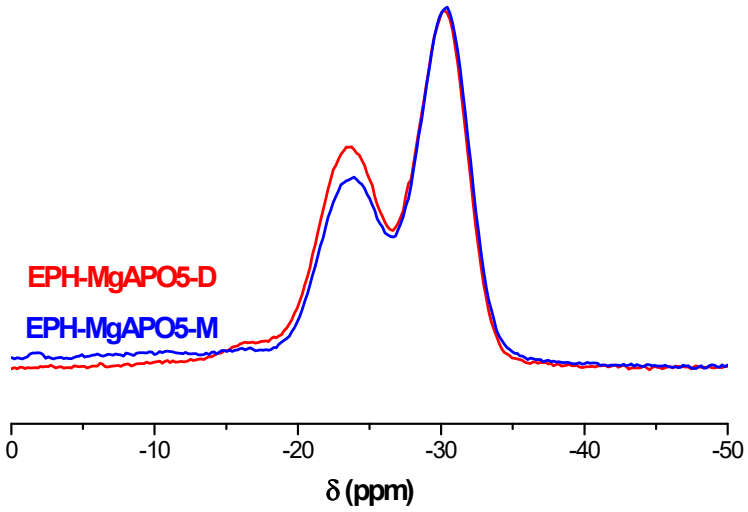

Figure S7. ^{31}P NMR spectra of MgAPO-5-M (top, blue line) and MgAPO-5-D (bottom, red line).

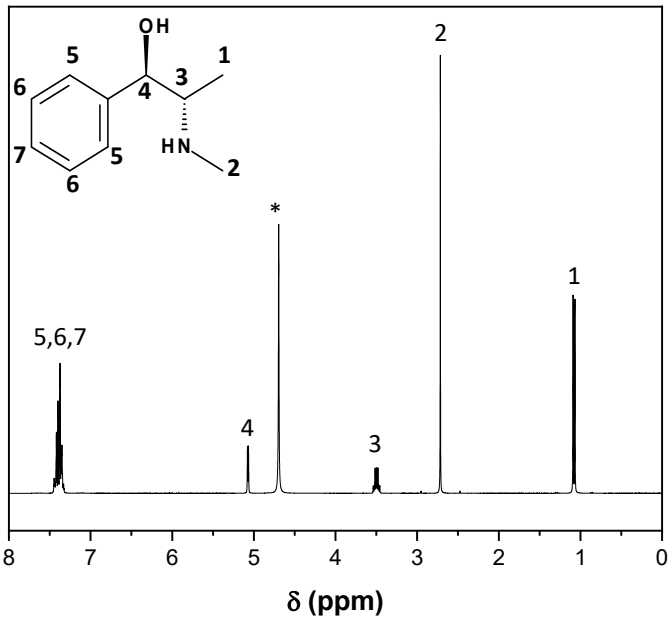

Figure S8. Liquid ^{1}H NMR spectra of ephedrine hydrochloride in D2O; * indicates band assigned to water.

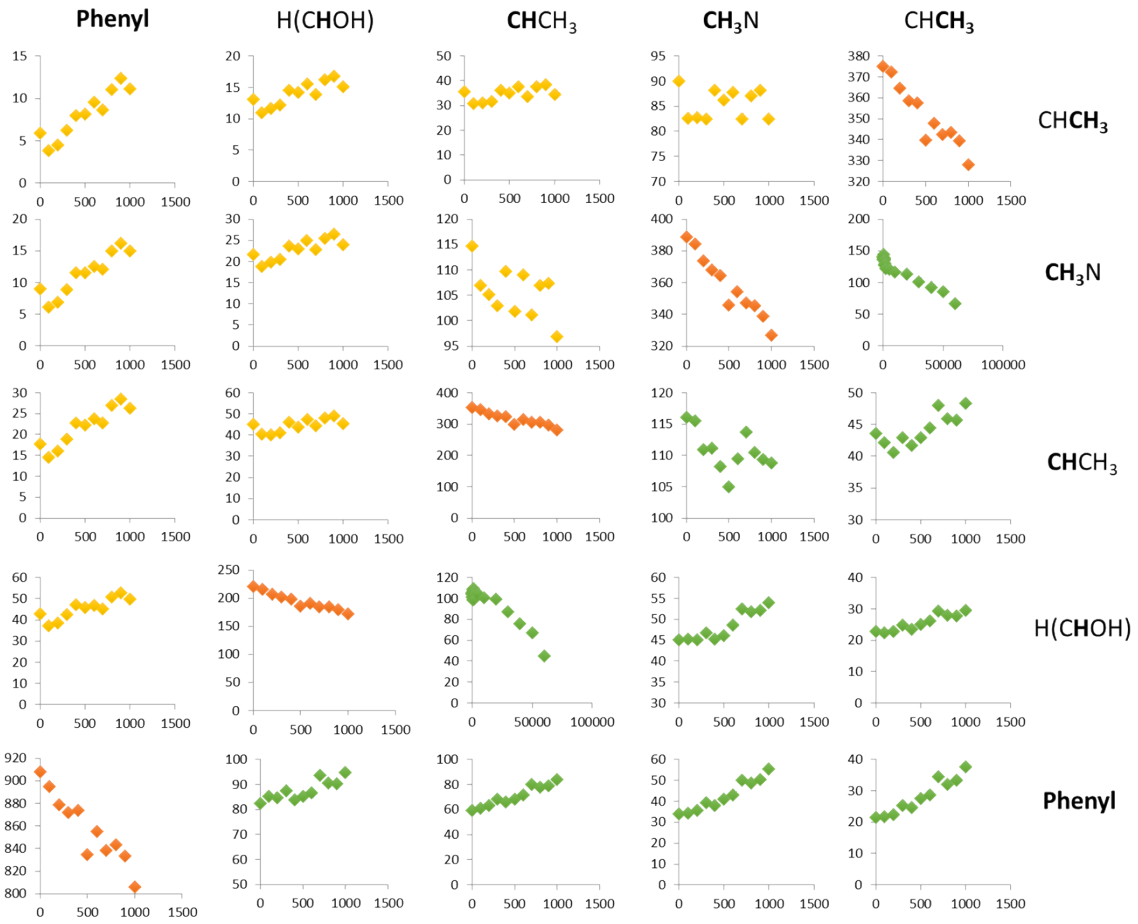

Figure S9. Measured peak intensities as a function of spin diffusion mixing time varying from 0 to 1000 microseconds for the 25 peaks observed in the 2D spin diffusion experiment for MgAPO-5-M.