ICP-2: A New Hybrid Organo-Inorganic Ferrierite Precursor with Expanded Layers Stabilized by π-π Stacking Interactions

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Supporting Information

MAS NMR details

Solid State MAS NMR were collected at room temperature on a Bruker AV-400-WB equipped with a 4 mm triple probe, using ZrO rotors, while spinning the sample at 10 kKHz in all cases.

^{27}Al (104.26 MHz) MAS NMR spectra are accumulated for 8k scans with a relaxation time of 0.5 s. The spectral width is 50 kHz, and π/30 pulses of 0.6 μs are used. $Al(SO_4)_2(NH_4).12H_2O$ is used as a secondary reference (-0.4 ppm) with respect to $Al(NO_3)_3$ 0.1M as primary reference.

For ^{29}Si MAS NMR spectra (79.49 MHz), π/2 pulses of 4.5μs. The spectral width is 40 kHz, and the relaxation time is 60 s. Kaolin (-91.2 ppm) is used as a secondary reference relative to TMS as primary reference.

For Cross-Polarization ^{1}H-^{13}C MAS NMR spectra (400.13 MHz for ^{1}H, 100.61 MHz for ^{13}C), the band width is 35 KHz, using a ^{1}H excitation pulse of 2.75 μs, a contact time of 3 ms and a relaxation time of 4 s, with tppm15 ^{1}H decoupling at 80 KHz. Adamantane (CH_2 29.5 ppm) is the secondary reference relative to TMS as primary reference.

^{19}F MAS NMR uses a 2.5mm probe. 90º pulses of 2μs are used, with a relaxation time of 120s, while spinning the sample at 20 KHz. Na_2SiF_6 (-152.46 ppm) is used as secondary reference relative to $CFCl_3$ as primary reference.

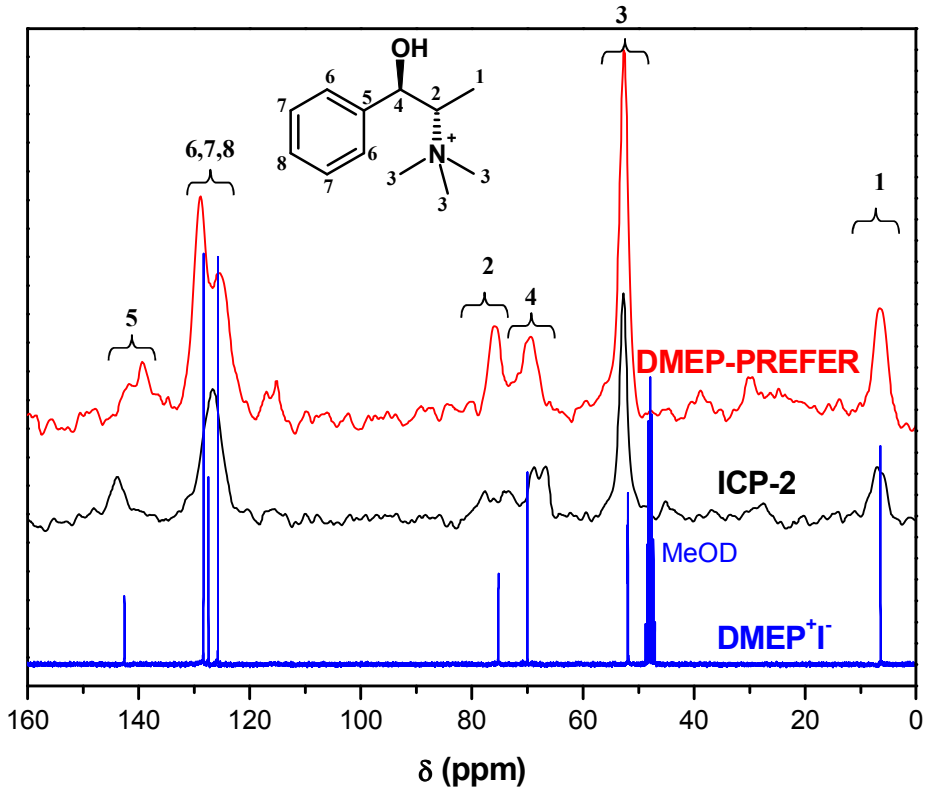

Figure S1. ^{13}C CP MAS NMR of as-made ICP-2 (black line) and DMEP-PREFER (red line) materials; liquid ^{13}C NMR of DMEP iodide (in MeOD) is also shown for comparison (signals at 49.2 ppm are due to MeOD).

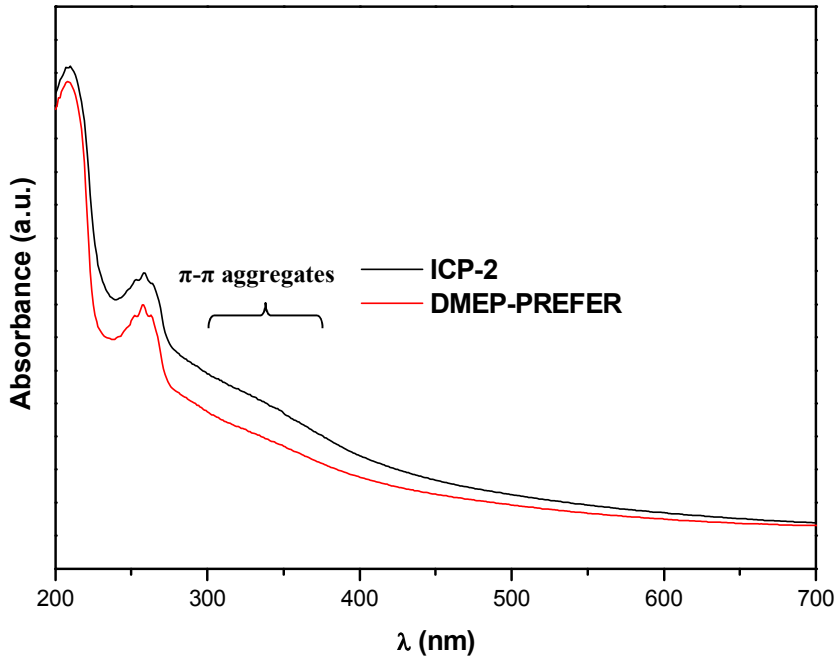

Figure S2. Diffuse-reflectance UV absorption of as-made ICP-2 (black line) and DMEP-PREFER (red line) materials.

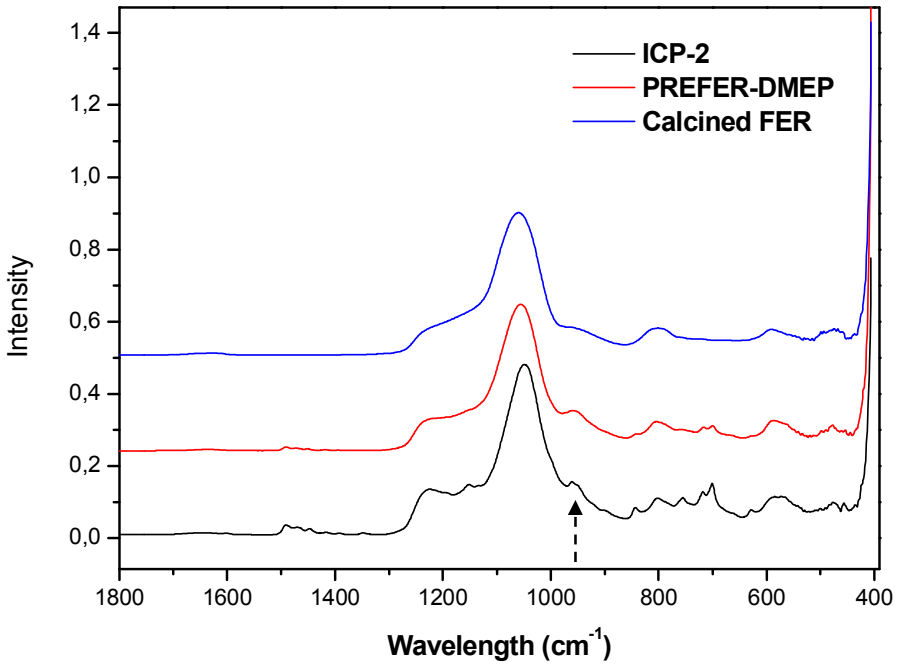

Figure S3. ATR-IR spectra of ICP-2 (bottom), PREFER-DMEP (middle) and calcined FER (top), highlighting the band at 960 cm^{-1} characteristic of silanol groups.

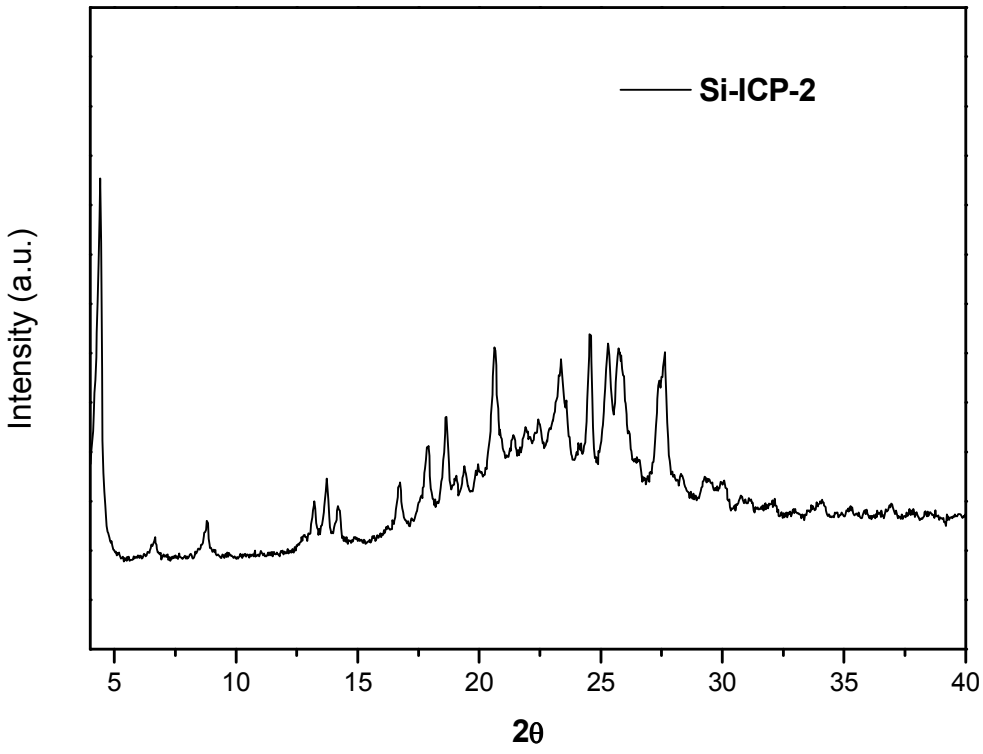

Figure S4. XRD pattern of all-silica ICP-2.

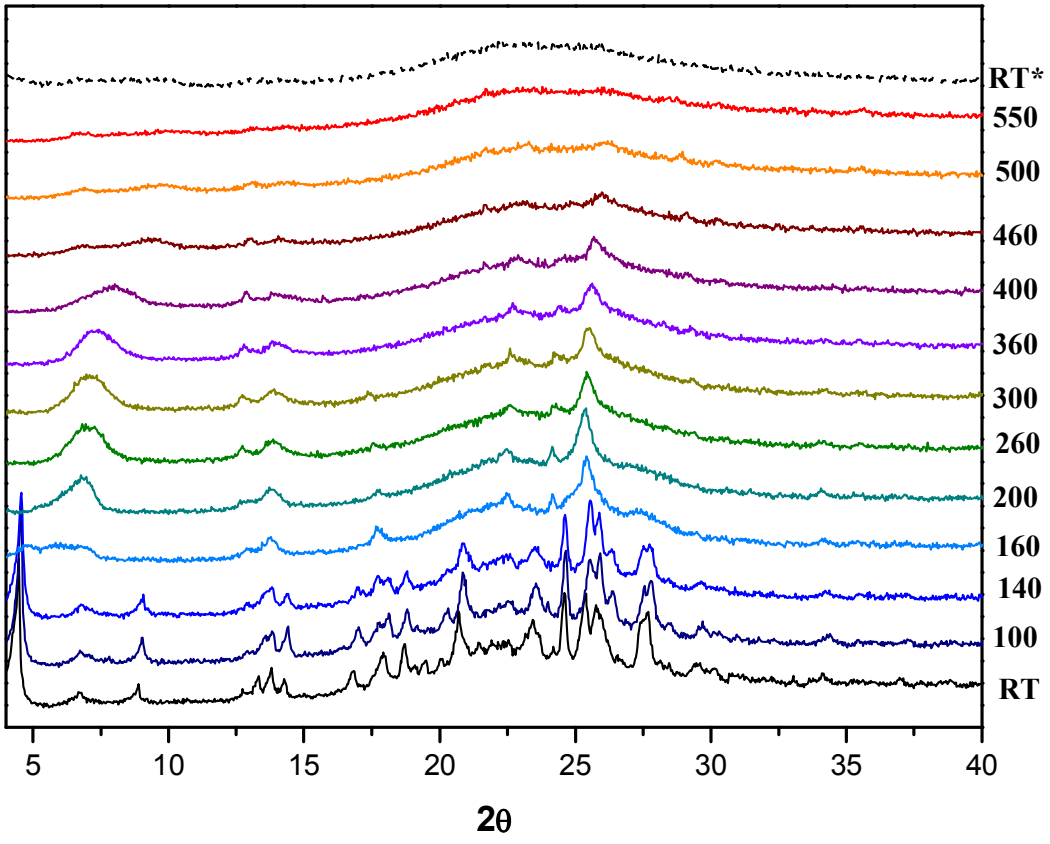

Figure S5. In-situ XRD patterns of the pure silica-version of ICP-2(Si) at increasing temperatures; * denotates the final material after cooling at room temperature.

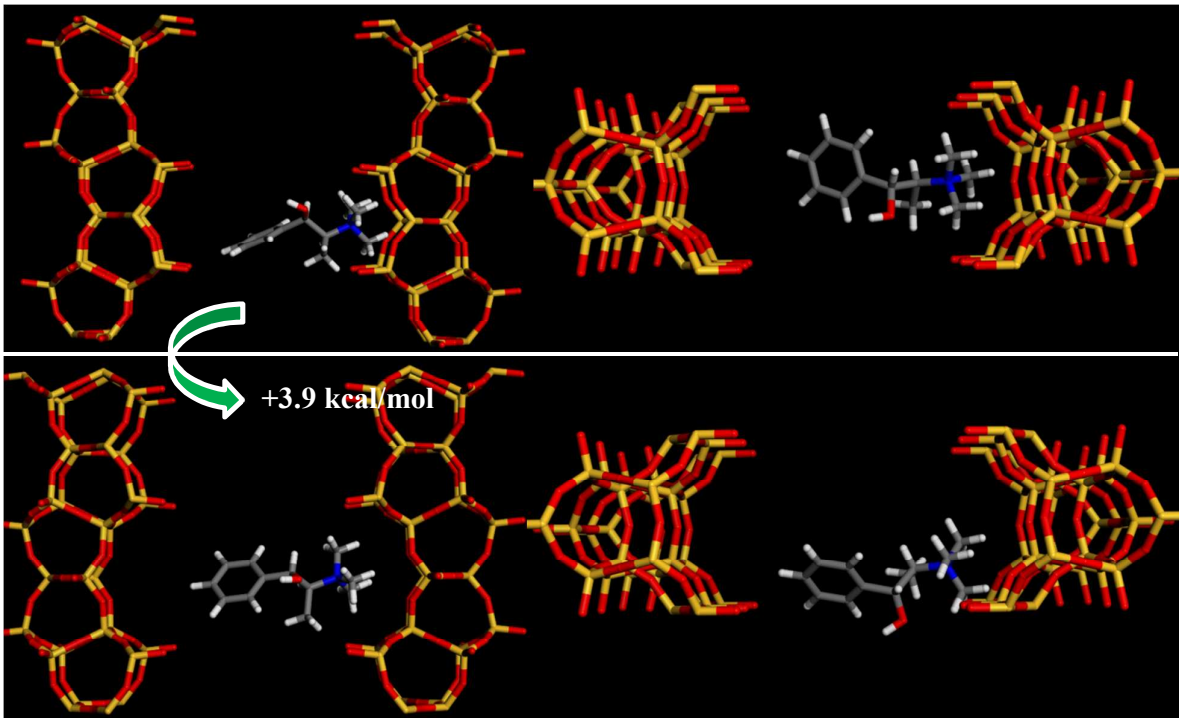

Figure S6. Two views (left and right) of the location of one DMEP cation in the FER pseudo-cavities (top) or in the pseudo-10R-channels (bottom).

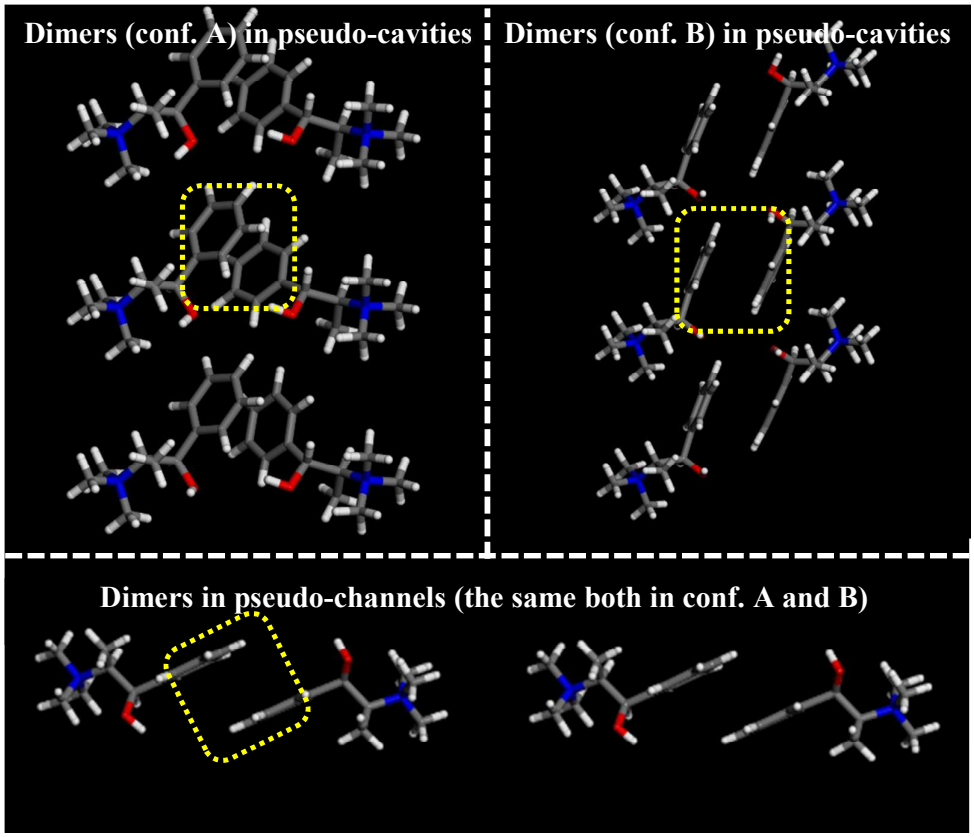

Figure S7. Details of the π-π stacking of DMEP cations in ICP-2.

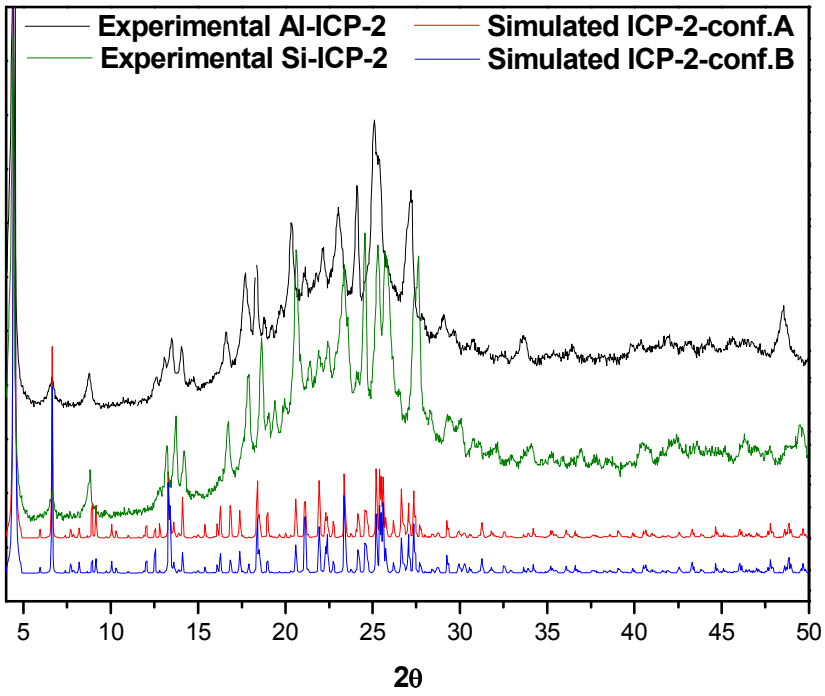

Figure S8. Comparison of the experimental XRD pattern of ICP-2 (black and green lines) and of the ICP-2 models (in conf. A, red line, or conf. B, blue line).