

Synthesis of Mesoporous Palladium with High Thermal Stability and Tunable Pore Size for Hydrogen Isotope Storage

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Supplemental Information: Figures S1-S5, Table S1, description of video

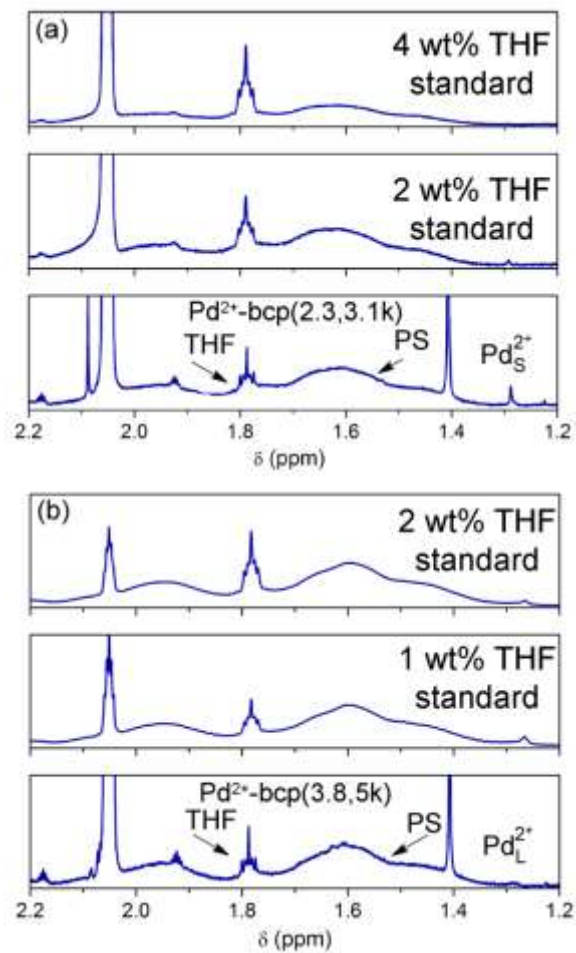


Figure S1. NMR spectra of bcp(2.3,3.1k) (a) and bcp(3.8,5k) (b) in d_6 -acetone. Top two spectra in (a) and (b) are calibration standards and bottom spectra are Pd²⁺-loaded LLC pastes subsequent to THF removal, immediately prior to metal salt reduction

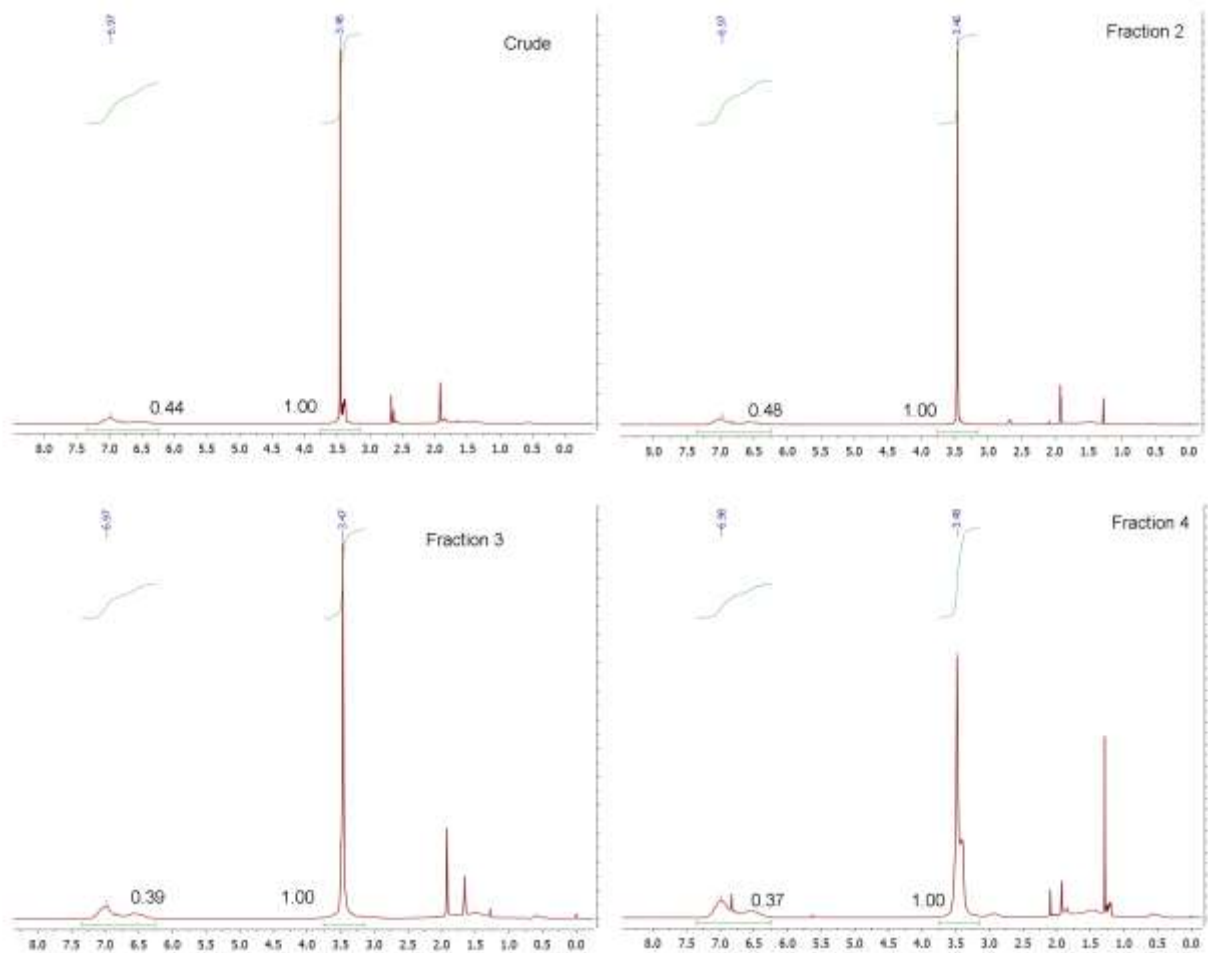


Figure S2. NMR spectra of the THF wash subsequent to Pd₅ synthesis (crude) and fractions two, three and four from THF elution on a silica column (60 mL fractions), in d₆-acetone. Peaks between 7.35 and 6.25 ppm are from the poly(styrene) block, and peaks between 3.75 and 3.15 are from the poly(ethylene oxide) block.

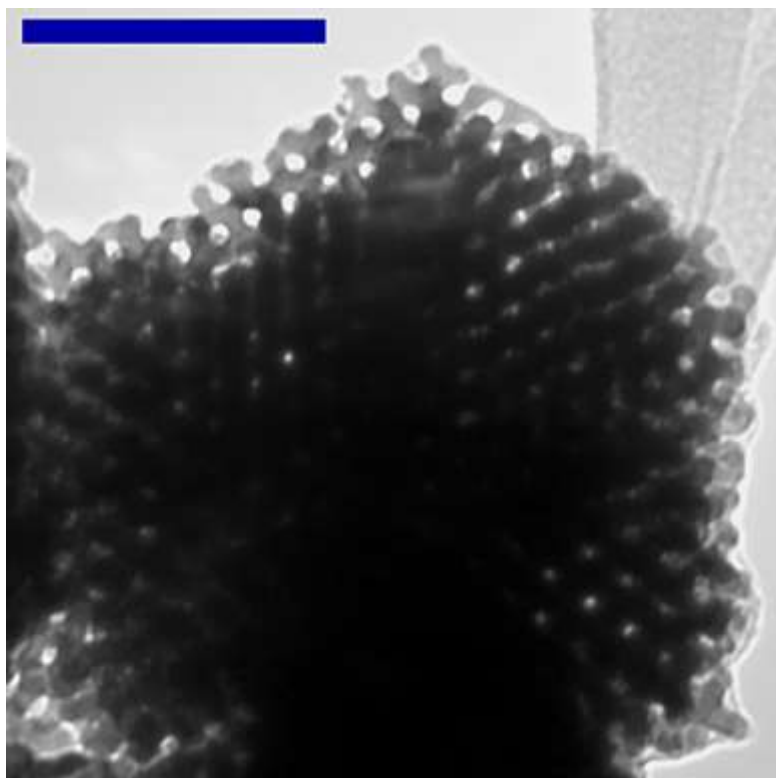


Figure S3. Porous Pd (Pd_s) synthesized from bcp(2.3,3.1k) purified after an initial use and recycled. (scalebar is calibrated to 100 nm)

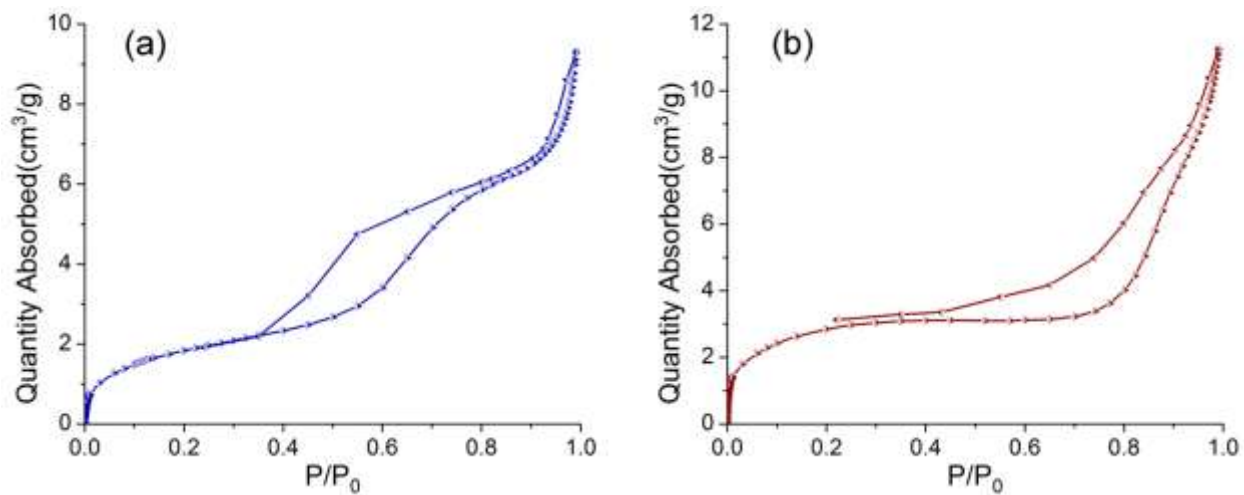


Figure S4. Nitrogen adsorption isotherms collected at 77K for Pd_S (a) and Pd_L (b)

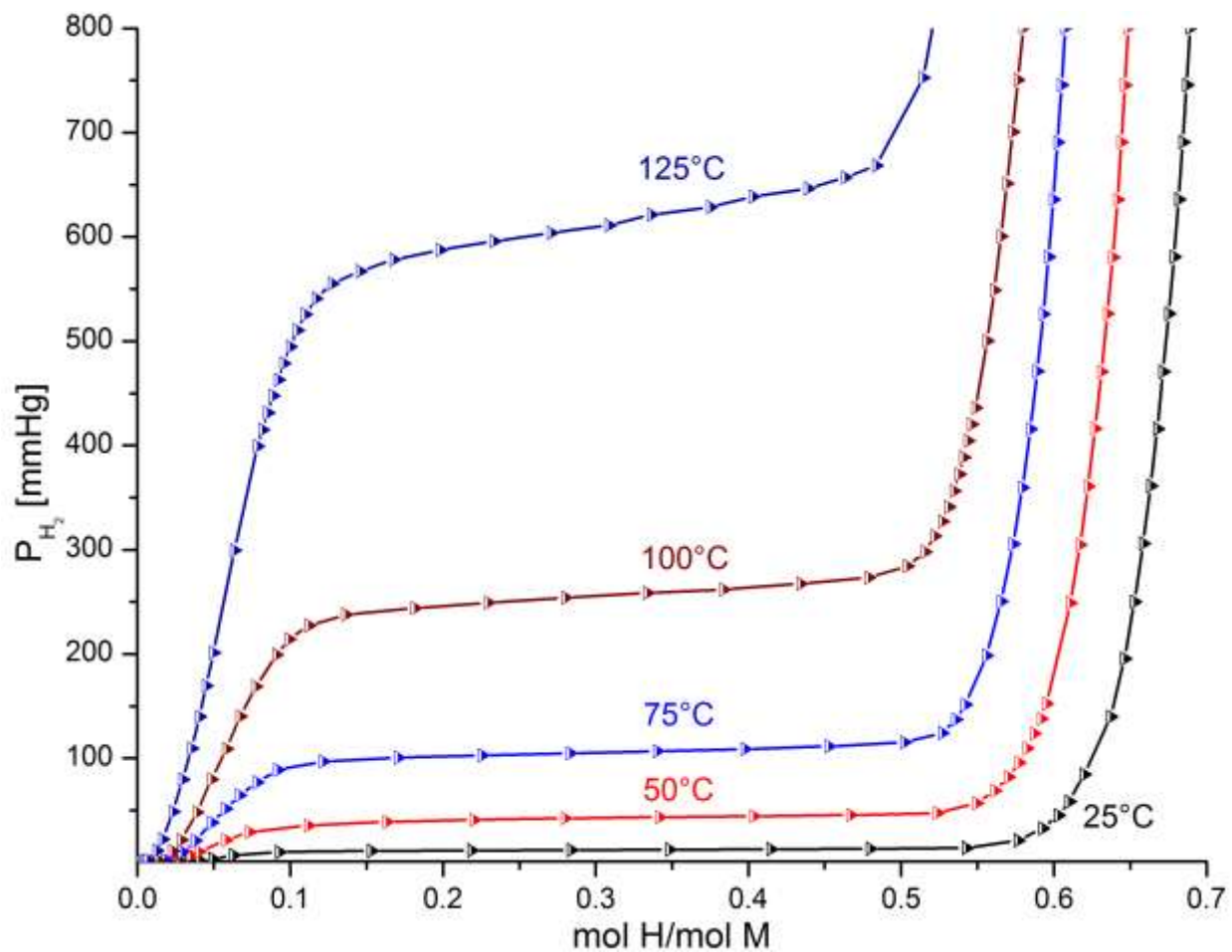


Figure S5. Linear H₂ storage isotherms at temperatures from 25°C to 125°C

Table S1. Rietveld refinement results

Sample 3.8,5k

Space group (No.)	F m -3 m (225)
Lattice parameters	
a=b=c/ Å	3.8909(3)
$\alpha=\beta=\gamma/^\circ$	90
V/ 10 ⁶ pm ³	58.90426
Pref. orientation direction/ hkl	1.00 0.00 0.00
Pref. orientation parameter	0.947(6)
R (Bragg)/ %	1.14
Crystallite Size/ Å	105.0

Sample 2.3,3.1

Space group (No.)	F m -3 m (225)
Lattice parameters	
a=b=c/ Å	3.8912(1)
$\alpha=\beta=\gamma/^\circ$	90
V/ 10 ⁶ pm ³	58.91829
Pref. orientation direction/ hkl	0.00 0.00 1.00
Pref. orientation parameter	1.015428
R (Bragg)/ %	1.17
Crystallite Size/ Å	198.4

Description of supplemental video: The video, encoded with the Windows Media Video 9 codec in Adobe Premier, shows the particle in Figure 8 during the first 6 s pulse to 250°C. The video is about 50% slower than real time. A drift correction algorithm written by Joshua D. Sugar and David B. Robinson of Sandia National Laboratories was applied, and is the cause of the shifting border.