
Supporting Information for *New Journal of Chemistry*

Comparison of Porous and Nonporous Materials for Methane Storage

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Experimental Details:

Solid state NMR was done on a Bruker DRX300 wide bore spectrometer equipped with a 7mm CP-MAS probe.¹ The operating frequencies are 300.13213 (¹H) and 75.47359 (¹³C). About 200 mg of finely ground powder was used to fully pack a 7mm Zirconia rotor. For all the samples proton decoupled ¹³C CP-MAS (Cross Polarization1 with Magic Angle Spinning)^{2,3} was performed at spinning rate of 5 KHz. TOSS (Total Suppression of Sidebands)⁴ was applied to remove the sidebands caused by spinning. The total time for data collection ranges from a few hours to overnight. The spectra were collected with 2K data points with 34 ms acquisition time and 2 s of relaxation delay. Data was zero-filled to 8K and a 5 Hz line-broadening was also applied. The external chemical shift reference was CO carbon of glycine at 176.03 PPM.

References:

1. A. Pines, M. G. Gibby and J. S. Waugh, *J. Chem. Phys.*, **1973**, *59*, 569.
2. J. Schaefer and E. O. Stejskal, In *Topics in ¹³C NMR spectroscopy*, vol. 3, (ed. G. C. Levy), pp. 283-324. Wiley, New York (1979).
3. E. R. Andrew, *Prog. N. M. R. spectrosc.*, **1971**, *8*, 1.
4. W. T. Dixon, *et. al.*, *J. Magn. Reson.* **1982**, *49*, 341.

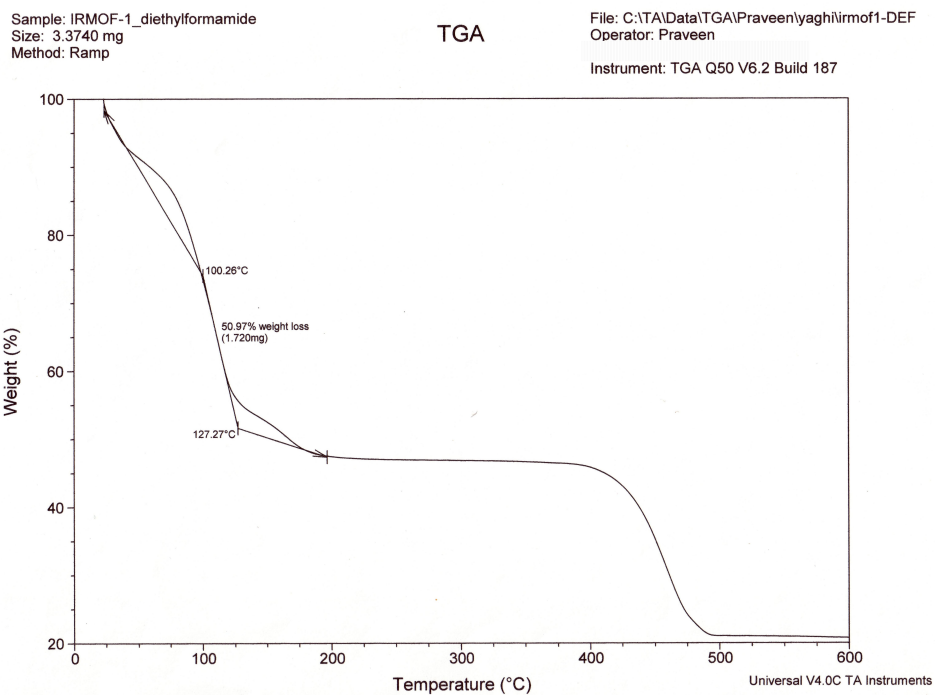


Fig. S1. Sample of **2** was heated gradually to 600 °C under inert atmosphere. A large and sharp weight loss of 50% of the original sample was observed below 200 °C, which attributes to the release of diethylformamide from the host cavity.

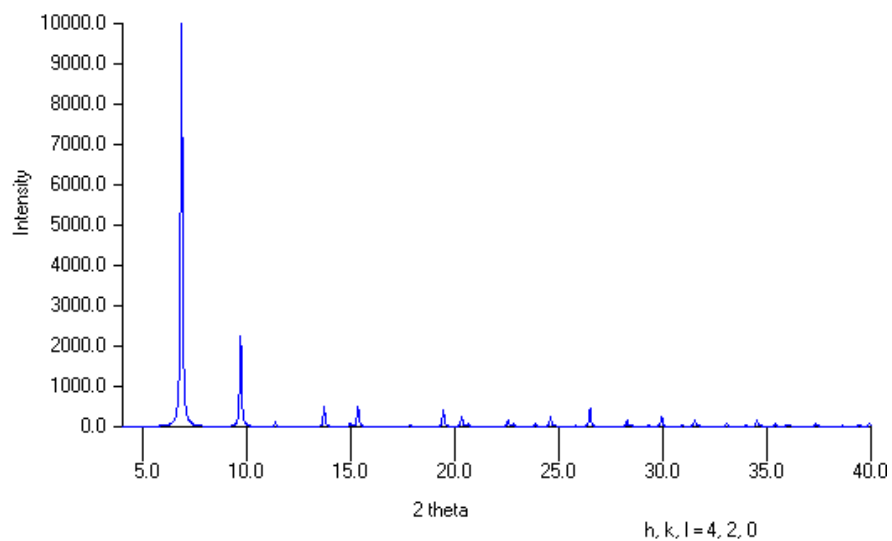


Fig. S2. Calculated powder pattern of **2** using the coordinates published by *Yaghi et. al.*, (Ref. 10a and 10d).

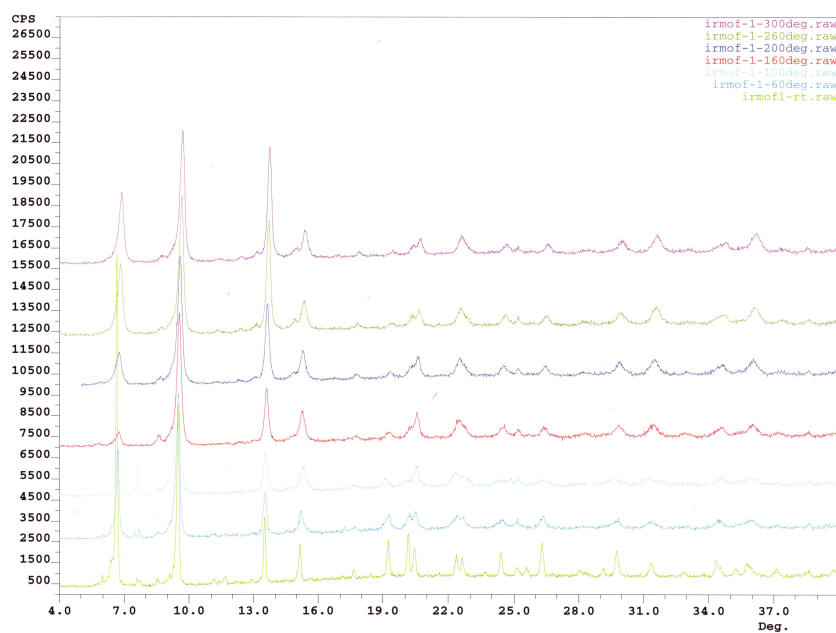


Fig. S3. Variable powder diffraction of **2** indicates the porous nature of the host molecule upon desolvation. Figure S1 and S2 represents the host network in sample **2** is identical to the reported IRMOF-1 (*Yaghi et. al.*, ref. 11a and 11d).

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Quantachrome Instruments
Quantachrome Autosorb Automated Gas Sorption System Report
Autosorb 1 for Windows 1.51

File name: C:\QCdata\PhysData\AS651601.raw
Sample ID: TBC 4 Description: Calixerene TBC 4
Comments:
Operator: jwb Sample weight: 0.083 g
Analysis gas: Nitrogen X sect. area: 16.2 Å²/molec Non-ideality: 6.58e-05
Adsbate (DRP): Nitrogen Bath Temp.: 77.30
Outgas Temp: 120.0 °C Outgas Time: 12.0 hrs Analysis Time: 67.6 min
P/Po tolerance: 0 Equil. time: 3 End of run: 05/16/2006 17:08
Station #: 1 PC sw. version: 1.51 TempComp: Off
MULTIPOINT BET

P/Po	Volume [cc/g] STP	1/(W((Po/P)-1))
5.2632e-02	0.1271	3.498E+02
7.7975e-02	0.2025	3.341E+02
1.0291e-01	0.2604	3.525E+02
1.5275e-01	0.4489	3.214E+02
2.0291e-01	0.5949	3.424E+02
2.5268e-01	0.8361	3.236E+02
3.0239e-01	1.1299	3.069E+02

Area = 1.589E+01 m²/g

Slope = -1.361E+02

Y - Intercept = 3.552E+02

Correlation Coefficient = 0.763987

C = 6.169E-01

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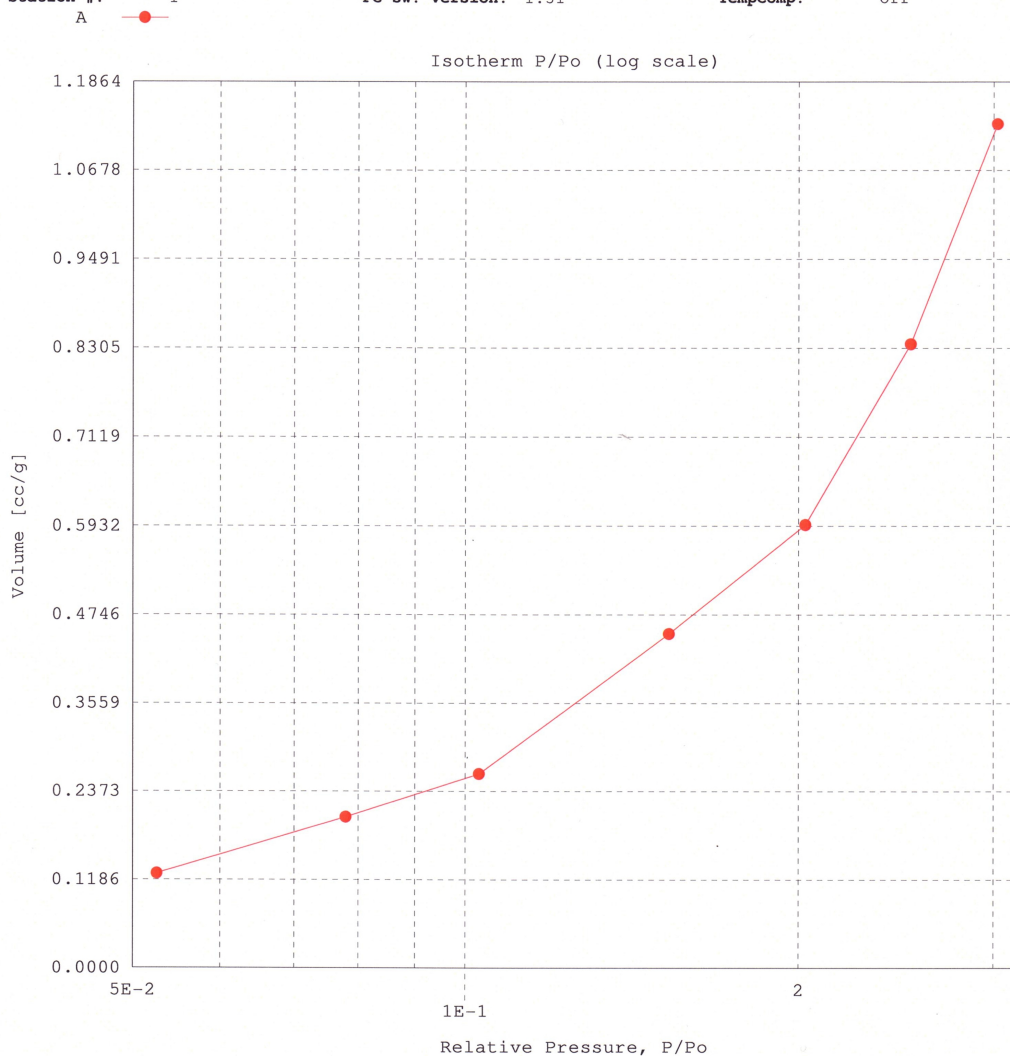
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Operator: jwb Sample weight: 0.083 g
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Isotherm

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Comments:			
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Outgas Temp:	120.0 °C	Outgas Time:	12.0 hrs
P/Po tolerance:	0	Equil. time:	3
Station #:	1	PC sw. version:	1.51
		Non-ideality:	6.58e-05
		Analysis Time:	67.6 min
		End of run:	05/16/2006 17:08
		TempComp:	Off



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Operator:	jwb	Bath Temp.:	77.30
Analysis gas:	Nitrogen	Outgas Time:	12.0 hrs
Adsorbate (DRP):	Nitrogen	Equil. time:	3
Outgas Temp:	120.0 °C	PC sw. version:	1.51
P/Po tolerance:	0	Non-ideality:	6.58e-05
Station #:	1	Analysis Time:	67.6 min
		End of run:	05/16/2006 17:08
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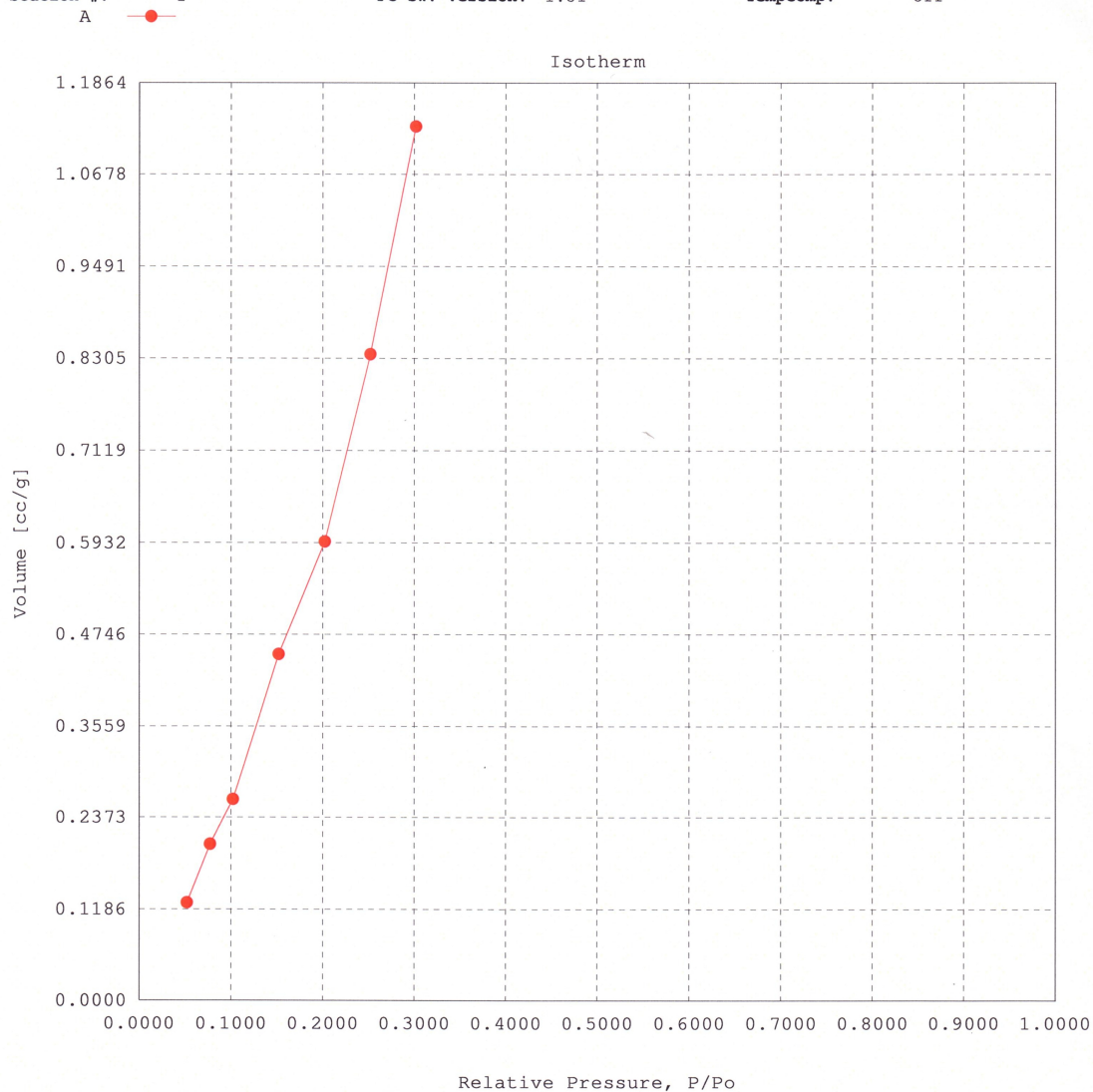


Fig. S4. BET measurement on sample 1 at 77 K and 1 atm pressure indicating the low surface area of the material.