Supplementary Material

for

Interconnected Water Channels and Isolated Hydrophobic Cavities in a Calixarene-Based, Nanoporous Supramolecular Architecture**

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Thermal analysis

DSC measurements were run on a TA 2920 calorimeter, samples of **3** (approximately 6 mg) were sealed in aluminium pans and heated at a rate of 1ºC/min under purified N_2 flow. TGA measurements were performed on a Mettler TG50 analyzer under purified N_2 flow heating at 0.5 ºC/min.

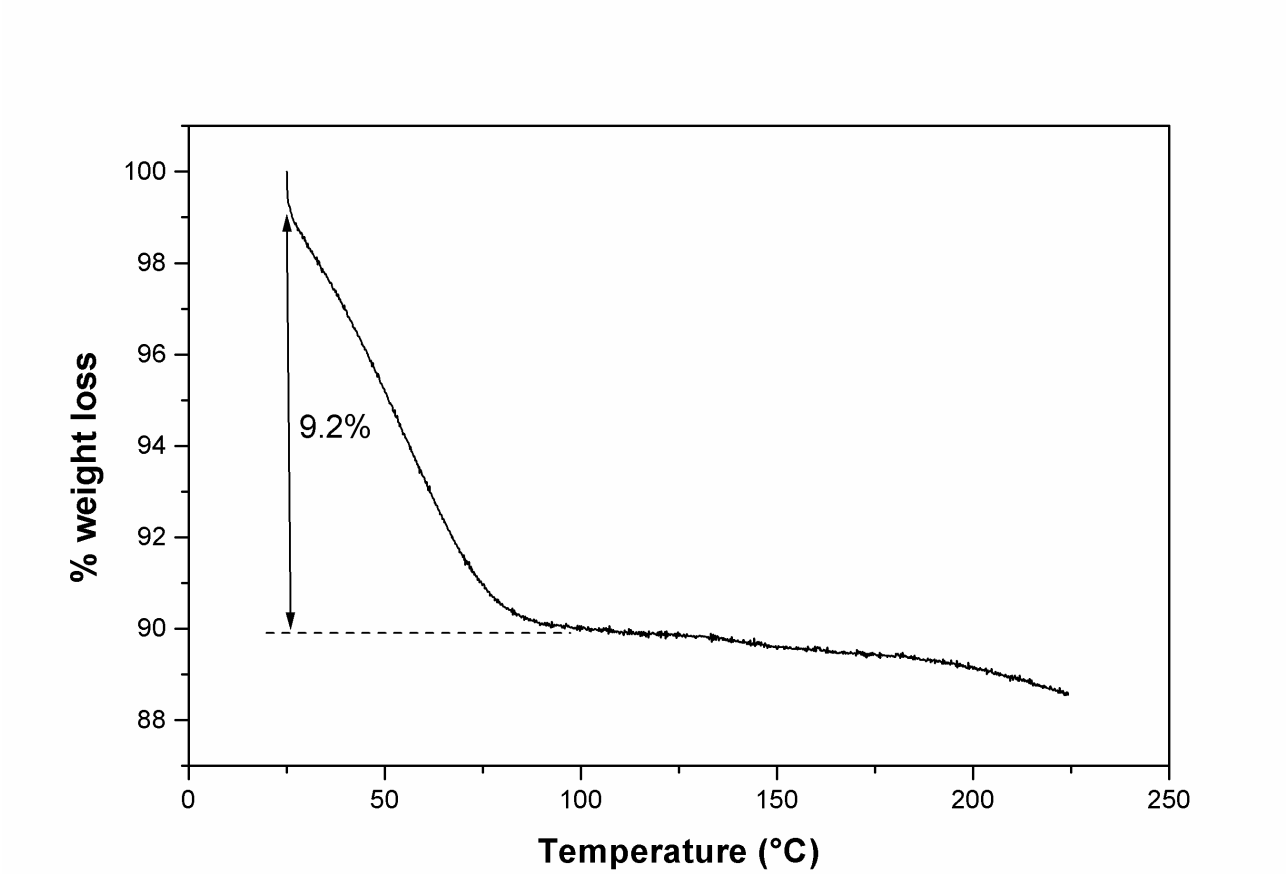

Figure S1. TGA curve of a crystalline sample of **3**, showing the weight loss due to the release of water from the channels (heating rate 0.5°C/min purified N_2 flow).

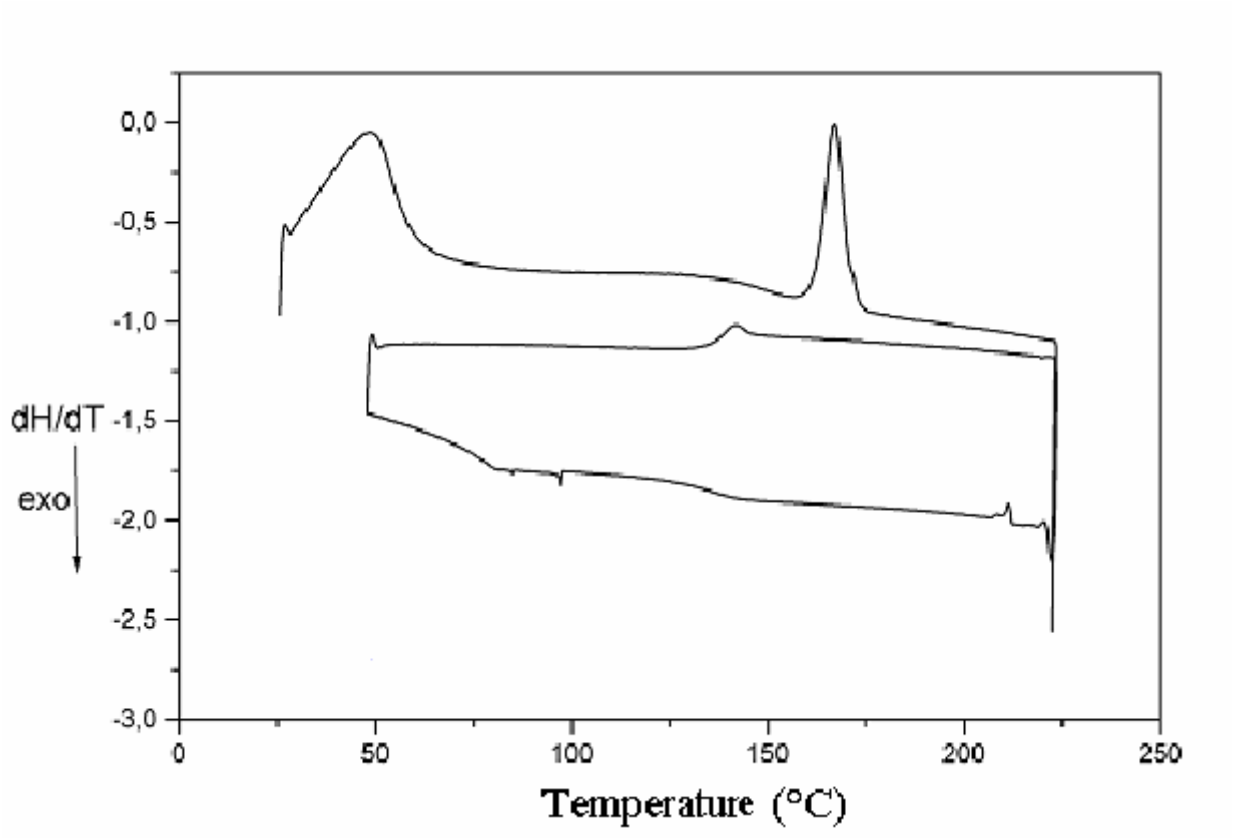

Figure S2. DSC profile of a sample of **3** showing the melting of the crystal.

X-ray powder diffraction and Rietveld Analysis

Crystalline powder samples were obtained from a solution of **3** in $CHCl_3$ and ethyl acetate. 0.3 mm Lindemann capillaries were filled in air with the as prepared crystalline powder. 15 mg of crystalline powders were treated at 50°C in vacuum for 8 h and stored in a dry box under N_2 atmosphere. 0.3 mm Lindemann capillaries were filled with the dried samples and sealed in a dry box under nitrogen atmosphere. X-ray powder diffraction profiles have been recorded using CuKα radiation (λ =1.5418 Å) by means of a Bruker D8 diffractometer equipped with a Goebel mirror. Data reduction was performed using the program GUFI.[1] The background was modelled manually using GUFI. Rietveld refinement was accomplished with the program GSAS.[2] The peak-profile was described by a pseudo-Voigt function, in combination with a special function that accounts for the asymmetry due to axial divergence.[3] No absorption correction was applied to the data. The atomic parameters were taken from the structure model obtained from the single crystal X-ray diffraction data and were not refined. The refined lattice paramater is *a*= 36.239(2) Å.
Final indices are R_p=0.053, $_wR_p$=0.072.

[1] R. E. Dinnebier, L. W. Finger, *Z. Krist. Suppl.* **1998**, *15*, 148; available at www.fkf.mpg.de/xray/html/body_gufi_software.html.

[2] A. C. Larson, R. B. Von Dreele, *GSAS - General Structure Analysis System,* Los Alamos National Laboratory, Los Alamos, USA, **2001**. LANL Report LAUR 86-748; available by anonymous FTP from mist.lansce.lanl.gov.

[3] a) P. Thompson, D. E. Cox, J. B. Hastings, *J. Appl. Cryst.*. **1987**, *20*, 79; b) L. W. Finger, D. E. Cox, A. P. Jephcoat, *J. Appl. Cryst.* **1994**, 27, 892.

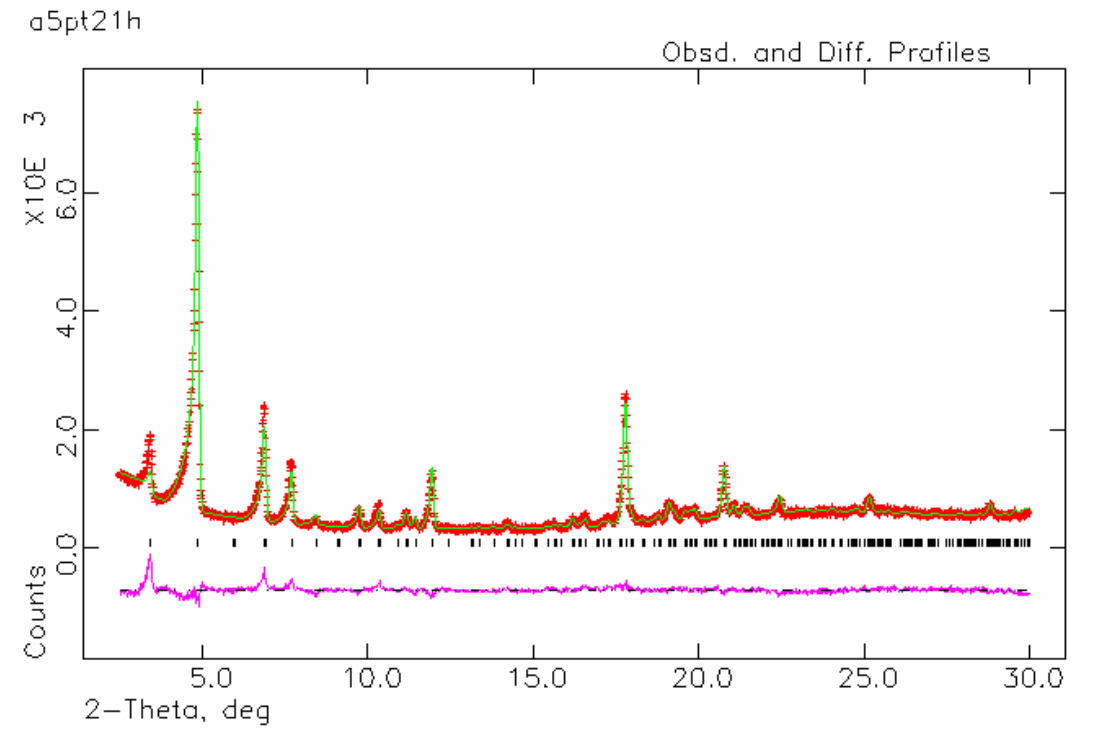

Figure S1. Scattered intensity of calixarene compound **3** as a function of the diffraction angle 2θ. Shown are the observed (red) and calculated X-ray powder diffraction pattern (green). The difference curve between observed and calculated profiles (magenta) and reflection markers (vertical bars).