Electronic Supplementary Information

Selective Alcohol Adsorption in a Uniformly Ordered Array of Lipophilic Mesopores by a Giant Macrocycle

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General

All reagents were reagent-grade and used without purification. Melting points were measured on a Yanaco MP-S3 melting point apparatus and are uncorrected. The ^{1}H NMR spectra were recorded on a Jeol JNM-AL300 spectrometer, operating at 300 MHz. The ^{13}C NMR spectra were measured on a Bruker DRX 600 spectrometer at a measurement frequency of 125 MHz. The chemical shifts are given in ppm downfield from the ^{1}H and ^{13}C signals of tetramethylsilane. IR spectra were recorded on a Nicolet Magna 720 FT-IR spectrometer. The FAB mass spectra were obtained on a Jeol JMS-70 mass spectrometer with m-nitrobenzyl alcohol as a matrix. Elemental analyses were performed at the Center of Elementary Analysis affiliated with Faculty of Science, Kyushu University. The X-ray powder diffraction data were collected on a Rigaku MultiFlex diffactometer over the 2θ range of 5–60° with using a copper radiation source.

Synthesis

Preparation of macrocycle **1** was previously reported[1] and obtained as follows: Under Ar atmosphere, a solution of tris(2-aminoethyl)amine (0.029 g, 0.20 mmol) in dry MeOH (100 mL) and a solution of *trans*-[bis(4-formylphenylethynyl)bis (triethylphosphine)]platium (0.21 g, 0.30 mmol) in dry CH_2Cl_2 (100 mL) were simultaneously added to a mixture of solvent of MeOH (200 mL) and CH_2Cl_2 (200mL) at room temperature with stirring. After stirring for 2 days, the mixture was concentrated to the volume of one-half. The precipitate was filtered and used without further purification. Yield: 0.146 g (62%); Mp 217 °C (dec.); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.43 (s, 12H, CH=N), 7.19 (d, J = 8.4 Hz, 24H, Ar-H), 6.72 (d, J = 8.4 Hz, 24H, ArH), 3.46 (br, 24H, CHNCH_2CH$_2$), 2.76 (br, 24H, CHNCH$_2$CH_2), 2.06-2.24 (m, 72H, PCH$_2$), 1.15-1.34 (m, 108H, CH$_3$); ^{13}C NMR (125 MHz, $CDCl_3$): δ 161.6 (s, C=N), 132.8 (Ph), 132.8 (Ph), 130.5 (Ph), 128.0 (Ph), 111.0 (t, $^{2}J_{PC}$ = 15 Hz, PtC≡C), 110.5 (PtC≡C), 59.9 (NCH$_2$), 56.8 (NCH$_2$), 16.5 (t, $^{1}J_{PC}$ = 17 Hz, PCH$_2$), 8.52 (s, CH$_3$); IR (KBr): ν_{max} 1640 (νC=N), 2110 (νC≡C) cm^{-1}; FABMS m/z: 4505.78 ([M^{+}], $C_{204}H_{288}N_{16}P_{12}Pt_6$); Anal. Calcd for $C_{204}H_{288}N_{16}P_{12}Pt_6$: C, 54.37; H, 6.44; N, 4.97; Found: C, 54.18; H, 6.43; N, 5.00.

Gas sorption

Nitrogen gas and alcohol sorption of **1** was performed on an automatic volume

adsorption apparatus (Bel Belsorp-18-plus). Prior to measurement, macrocycle **1** was dried at 70 °C for 7 h under reduced pressure of 3×10^{-4} kPa, then gradually cooled to 30 °C. The dried sample and sample tube were weighed precisely and transferred to the analyzer. The sorption isotherms were collected in a relative pressure range from 0.002 to 0.95. For nitrogen sorption of **1**, liquid nitrogen bath was used for controlling the temperature at 77 K. All of the measurements were analyzed with a Belmaster (ver. 5.3.5.2) implemented in the apparatus. The specific surface area and pore size distribution were obtained by the Brunauer-Emmet-Teller (BET) method[2] and the Cranston-Inkley (CI) method[3] using nitrogen gas sorption. The isosteric heat of sorption for alcohol was obtained by the Clausius-Clapeyron equation[4] using alcohol isotherms at 228 K and 308 K.

References

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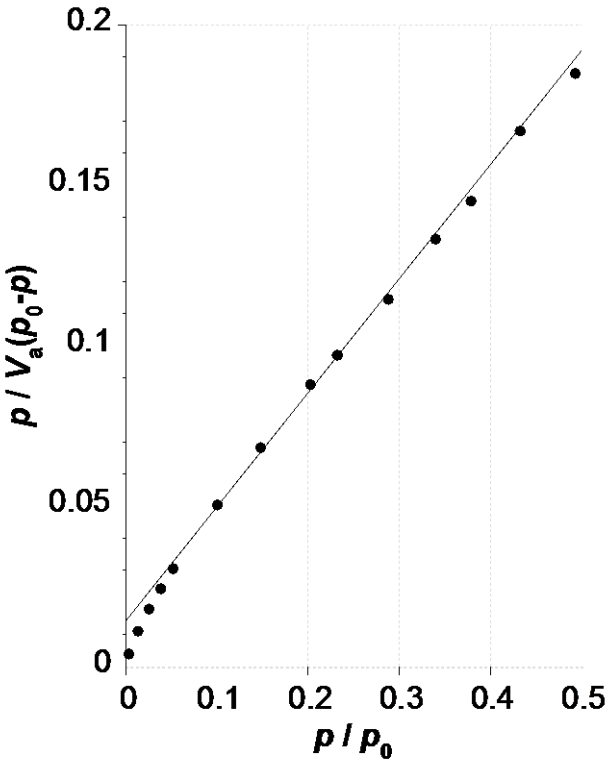

Fig. S1. BET plot calculated from nitrogen gas adsorption of **1** at 77 K. Linear line was obtained by least square method using data, where relative pressure is in the range of 0.052−0.288.