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Supporting Information

Self-Assembly of Magnesium-Seamed Hexameric Pyrogallol[4]arene Nanocapsules

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METHODS

Materials and Characterization. All solvents and chemicals were obtained from commercial sources and used without further purification. The single crystal X-ray diffraction data was collected on a Brucker Apex II diffractometer at a temperature of 100 (2) K using CuKα (1.54056Å) radiation incotec Microfocus II. The structure was solved and refined using SHELX and Xseed[1] as the interface. Internal void volumes of each nanocapsule were calculated using MSRoll with a probe radius of 1.25 Å.

Synthesis of *C*-propylpyrogallol[4]arene (PgC_3). Butyraldehyde (7.21 mL, 0.08 mol), and pyrogallol (0.08 mmol, 10 g) were mixed in 40 mL of 95% (v/v) ethanol with the addition of 3.5 mL of concentrated HCl. Thereafter, the mixture was refluxed at 110 ℃ for 24 hours. After cooling down, the precipitate was filtered, washed with cold 95% (v/v) ethanol and dried in vacuum. 5.4 g of white solid was prepared as the final product, PgC_3. Yield is 37.5%.

Preparation of 1 [$Mg_{24}(C_{40}H_{40}O_{12})_6(DMF)_4(H_2O)_{44}$]. *C*-propylpyrogallol[4]arene (PgC_3, 0.1 mmol, 72.0 mg) and $Mg(NO_3)$ $6H_2O$ (0.4 mmol, 102.6 mg) were dissolved in 1 mL of *N*,*N*-dimethylformamide (DMF) and 1 mL of acetonitrile (MeCN) with the addition of 100 μL of water and 27.2 mg of imidazole (0.4 mmol) in a 4 mL glass vial. The mixture was sonicated for 5 min to yield a black solution, and then heated at 100 ℃ overnight. Green crystals were then formed and collected for single crystal X-ray analysis.

Preparation of 2 [$Mg_{24}(C_{40}H_{40}O_{12})_6(H_2O)_{48}$ ·2py]. *C*-propylpyrogallol[4]arene (PgC_3, 0.1 mmol, 72.0 mg) and $Mg(NO_3)$ $6H_2O$ (0.4 mmol, 102.6 mg) were dissolved in 3

mL of acetonitrile (MeCN) with the addition of 100 μL of water and 100 μL of pyridine (1.2 mmol) in a 20 mL glass vial. The mixture was sonicated for 5 min to yield a dark grey suspension, and then heated at 100 ℃ overnight. Dark blue crystals were then formed and collected for single crystal X-ray analysis.

Supplementary Data

Table S1: Single crystal structure data of **1** and **2**.

	1	**2**
Molecular formula	$Mg_{24}(C_{40}H_{40}O_{12})_6(DMF)_4(H_2O)_{44}$	$Mg_{24}(C_{40}H_{40}O_{12})_6(H_2O)_{48}\cdot 2py$
Crystal system	monoclinic	triclinic
Space group	C2/m	P-1
Temperature (K)	100	100
a(Å)	28.2464(14)	26.113(2)
b(Å)	35.3404(15)	26.276(2)
c(Å)	21.6242(10)	27.960(2)
α(°)	90	93.634(4)
β(°)	118.889(2)	92.074(5)
γ(°)	90	90.160(5)
V(Å^3)	18899.9(15)	19133(3)
Z	2	2
Calculated desnity	1.024	1.000
Θ range of data collection(°)	3.19-58.90	2.38-56.93
Total unique reflections	9060	9665
Goodness of fit	2.159	2.047
R1 after merging	0.1615	0.2219
R(int)	0.0413	0.0896
wR2	0.4657	0.5144

References:

1. L. J. Barbour, *Journal of Supramolecular Chemistry*, 2003, **1**, 189-191.