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Supporting Information

A Twelve-Connected Porous Framework Built from Rare Linear Cadmium Tricarboxylate Pentamer

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Reagents were purchased commercially and used without further purification. Powder XRD patterns were obtained using a Bruker D8 Advance X-ray powder diffractometer with Cu *K*α radiation (λ = 1.54056 Å). Thermal stability studies were carried out on a TA-Q500 thermoanalyzer with a heating rate of 5 °C/min under nitrogen atmosphere. Gas adsorption experiments were performed on Micromeritics ASAP 2020 surface area and pore size analyzer. The sample was degassed at 230 °C for 2 days prior to the measurement.

Synthesis:

Synthesis of $[Cd_5(BTB)_4(H_2O)_4]_n \cdot (NEt_4)_{2n}$ (**1**): A mixture of 1,3,5-tris(4-carboxyphenyl)benzene (H_3BTB, 0.0146 g, 0.0333 mmol), $Cd(NO_3)_2 \cdot (H_2O)_4$ (0.0308 g, 0.1000 mmol) and $N(Et)_4 \cdot ClO_4$ (0.0230 g, 0.1000 mmol), 1,3-dimethylpropyleneurea (dmpu, 4 mL), and together with 1 mL water, placed in a 10 mL vial, was heated at 120 °C for 3 days, and then cooled to room temperature. Colorless crystals of **1** were obtained (yield: 20 % based on H_3BTB).

Synthesis of $[Cd_3(BTB)_2(H_2O)_2(dmpu)_2]_n$ (**2**): A mixture of H_3BTB (0.0146 g, 0.0333 mmol), $Cd(NO_3)_2 \cdot (H_2O)_4$ (0.0308 g, 0.1000 mmol) and $N(Et)_4 \cdot ClO_4$ (0.0230 g, 0.1000 mmol), and together with mixed dmpu (4 mL)/water (1 mL) solution, placed in a 20 mL vial, was heated at 120 °C for 3 days, and then cooled to room temperature. Colorless crystals of **2** were obtained (yield: 35 % based on H_3BTB).

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Table S1. Crystal Data and Structure Refinements for Complexes **1** and **2**.

	1	**2**
Chemical formula	$C_{108}H_{68}Cd_5O_{28}•C_{16}H_{40}N_2$	$C_{66}H_{58}Cd_3N_4O_{16}$
Formula Mass	2636.17	1500.39
Crystal system	Monoclinic	Triclinic
a/Å	35.859(7)	10.0514(6)
b/Å	17.020(3)	14.6745(8)
c/Å	27.824(6)	14.8435(8)
α/°	90.00	69.202(3)
β/°	122.73(3)	83.200(4)
γ/°	90.00	88.432(4)
Unit cell volume/Å^3	14285(7)	2032.1(2)
Temperature/K	150(2)	150(2)
Space group	*C*2/*c*	$P\bar{1}$
No. of formula units per unit cell, *Z*	4	1
No. of reflections measured	40888	13183
No. of independent reflections	12800	12201
R_{int}	0.0432	0.0636
Final R_1 values ($I > 2\sigma(I)$)	0.0682	0.0565
Final $wR(F^2)$ values ($I > 2\sigma(I)$)	0.2056	0.1318
Final R_1 values (all data)	0.0821	0.0891
Final $wR(F^2)$ values (all data)	0.2143	0.1455
Goodness of fit on F^2	1.049	0.955

$R_1 = \sum||F_o| - |F_c||/\sum|F_o|$, $wR = \{\sum w[(F_o)^2 - (F_c)^2]^2/\sum w[(F_o)^2]^2\}^{1/2}$.

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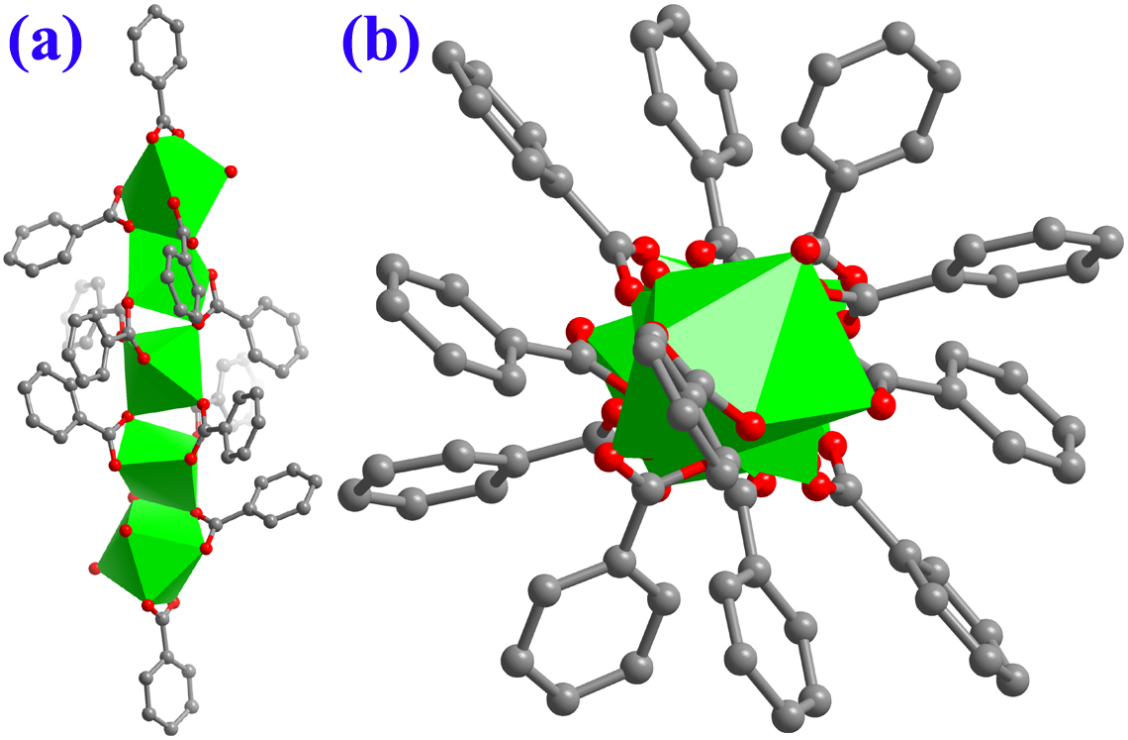

Figure S1. Lateral (a) and top (b) view of a linear $Cd_5(CO_2R)_{12}$ fragment showing each pentamer unit being connected to twelve tritopic BTB^{3-} ligands in **1**.

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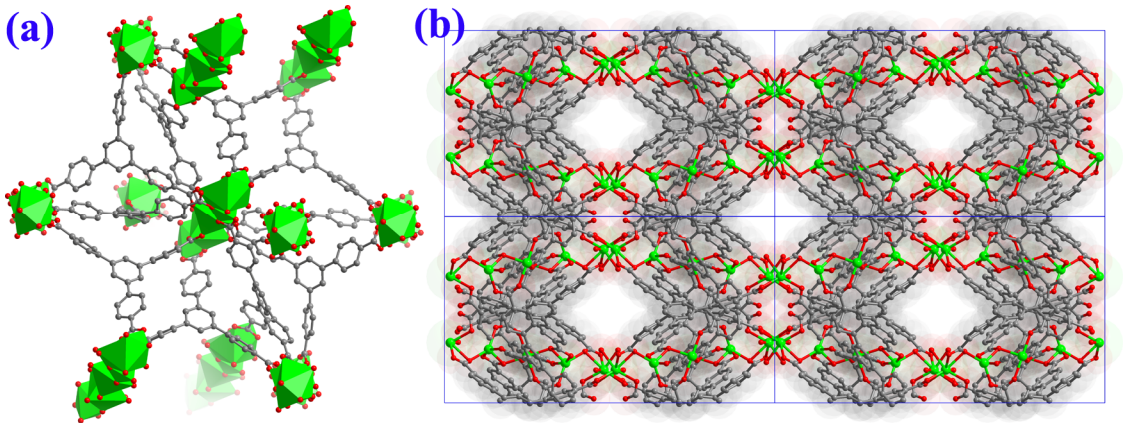

Figure S2. The 3D network connectivity of the linear $Cd_5(CO_2R)_{12}$ pentamers (a), and the channel packing viewed along [001] (b) in **1**.

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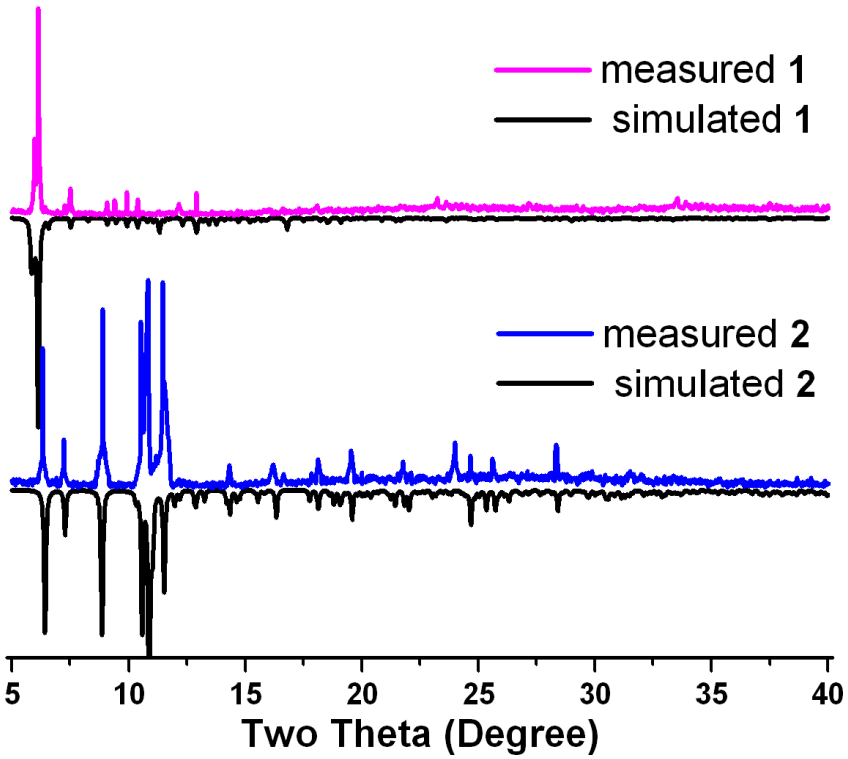

Figure S3. Simulated and measured XRD powder patterns for **1** and **2**, respectively.

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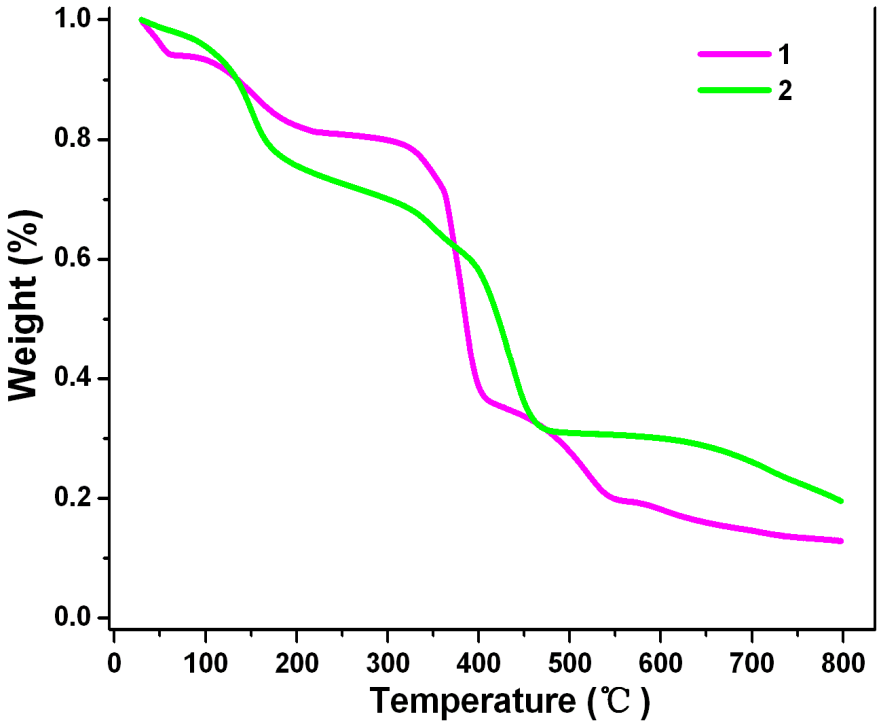

Figure S4. TGA curves of **1** and **2**, respectively.

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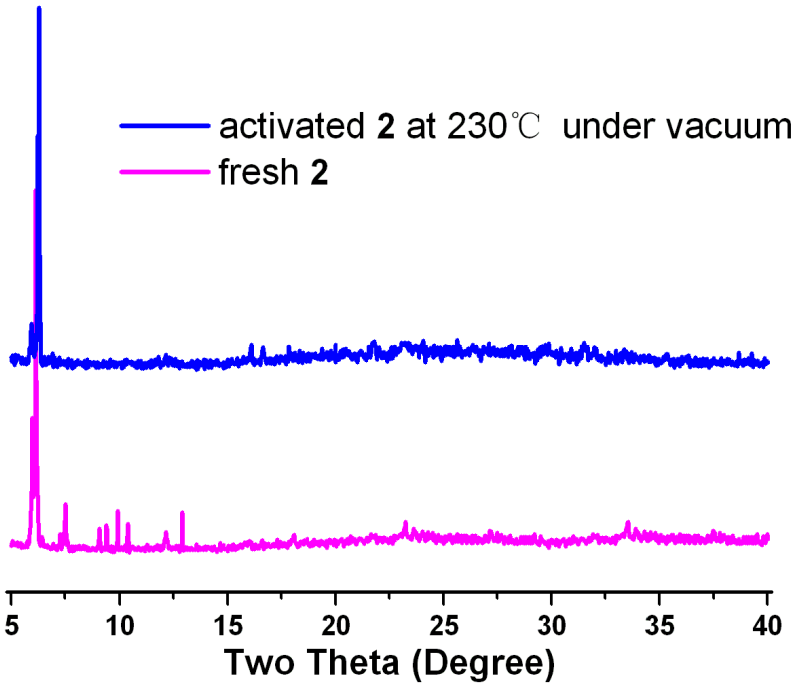

Figure S5. PXRD patterns from **1** before (magenta) and after (green) activated at 230 °C under vacuum.