Supplementary Material (ESI) for Chemical Communications

Supporting Information for *Chemical Communications*

Surface Functionalization of Metal-Organic Polyhedron for Homogeneous Cyclopropanation Catalysis

Weigang Lu, Daqiang Yuan, Andrey Yakovenko, and Hong-Cai Zhou*

Department of Chemistry, Texas A&M University, College Station, Texas 77843, USA

E-mail: zhou@mail.chem.tamu.edu

1. Materials and Methods

Commercially available reagents were used as received without further purification. Nuclear magnetic resonance (NMR) data were collected on a Mercury 300 MHz NMR spectrometer. Fourier transform infrared spectroscopy (FTIR) data were collected on a SHIMADZU IRAffinity-1 FTIR Spectrophotometer. Elemental analyses (C, H, and N) were obtained from Canadian Microanalytical Service, Ltd. ESI-MS experiments were performed on a Voyager DE-STR mass spectrometer (Applied Biosystems, Foster City, CA). Ultraviolet/Visible (UV-Vis) absorption spectra were recorded on a SHIMADZU UV-2450 UV-Vis Spectrophotometer. Thermogravimetry analyses (TGA) were performed under N_2 on a SHIMADZU TGA-50 Thermogravimetric Analyzer, with a heating rate of 5 °C min^{-1}. High performance liquid chromatography (HPLC) analysis was carried out on a SHIMADZU Prominence UFLC. Powder X-ray diffraction (PXRD) patterns were obtained on a BRUKER D8-Focus Bragg-Brentano X-ray Powder Diffractometer equipped with a Cu sealed tube (λ = 1.54178) at a scan rate of 0.2 s deg^{-1}, solid-state detector, and a routine power of 1400 W (40 kV, 35 mA).

2. X-ray Crystallography

Single crystal X-ray data (**MOP 1a**, **MOP 1b**, **MOP 2**, and **MOP 3**) were collected on a Bruker-AXS APEX-II CCD X-ray diffractometer equipped with a low temperature device and a fine-focus sealed-tube X-ray source (Mo-K_α radiation, λ = 0.71073 Å, graphite monochromated). Suitable single crystals were directly picked up from the mother liquor, attached to a glass loop and transferred to a designed cold stream of liquid nitrogen (110 K) for data collections. Raw data collection and reduction were done using APEX2 software.

Single crystal X-ray structure determination of **MOP 4** was performed at 100(2) K on the Advanced Photon Source on beamline 15ID-B in Argonne National Laboratory.

Structures were solved by direct method and refined by full-matrix least-squares on F^2 using *SHELXTL*[1]. Non-hydrogen atoms were refined with anisotropic displacement parameters during the final cycles. Organic hydrogen atoms were placed in calculated positions with isotropic displacement parameters set to 1.2 × U_{eq} of the attached atom. The solvent molecules are highly disordered, and attempts to locate and refine the solvent peaks were unsuccessful. Contributions to scattering due to these solvent

molecules were removed using the *SQUEEZE* routine of *PLATON*[2]; structures were then refined again using the data generated.

Crystal data for **MOP 1a**: $C_{50}H_{30}Cu_2O_{11}$, M = 933.82, blue prism, 0.22×0.20×0.17 mm^3, monoclinic, space group $C2/c$ (No. 15), a = 13.99831(9), b = 30.543(2), c = 27.992(2) Å, β = 101.349(3)°, V = 11721(1) Å^3, Z = 8, D_c = 1.058 g/cm^3, F_{000} = 3808, MoKα radiation, λ = 0.71073 Å, T = 110(2)K, $2\theta_{max}$ = 53.1º, 54362 reflections collected, 12065 unique (R_{int} = 0.0915). Final *GooF* = 0.800, *R1* = 0.0603, *wR2* = 0.1497, *R* indices based on 6088 reflections with I >2sigma(I) (refinement on F^2), 568 parameters, μ = 0.772 mm^{-1}. CCDC-797901.

Crystal data for **MOP 1b**: $C_{104}H_{72}Cu_4O_{24}S_2$, M = 2023.90, blue needle, 0.30×0.09×0.07 mm^3, tetragonal, space group $I4/m$ (No. 87), a = b = 25.160(3), c = 25.726(3) Å, V = 16285(3) Å^3, Z = 4, D_c = 0.825 g/cm^3, F_{000} = 4144, MoKα radiation, λ = 0.71073 Å, T = 110(2)K, $2\theta_{max}$ = 50.0º, 48691 reflections collected, 7171 unique (R_{int} = 0.1608). Final *GooF* = 1.026, *R1* = 0.0666, *wR2* = 0.1224, *R* indices based on 2789 reflections with I >2sigma(I) (refinement on F^2), 317 parameters, 0 restraints. μ = 0.585 mm^{-1}. CCDC-797902.

Crystal data for **MOP 2**: $C_{118}H_{86}Cu_4N_2O_{28}$, M = 2234.05, blue prism, 0.20×0.20×0.10 mm^3, triclinic, space group P-1 (No. 2), a = 14.852(3), b = 16.673(3), c = 17.458(6) Å, α = 110.385(4), β = 103.907(4), γ = 108.850(3)°, V = 3518.6(15) Å^3, Z = 1, D_c = 1.054 g/cm^3, F_{000} = 1148, MoKα radiation, λ = 0.71073 Å, T = 110(2)K, $2\theta_{max}$ = 52.0º, 27594 reflections collected, 13625 unique (R_{int} = 0.0733). Final *GooF* = 1.012, *R1* = 0.0710, *wR2* = 0.1361, *R* indices based on 5835 reflections with I I>2(I) (refinement on F^2), 574 parameters, 40 restraints. μ = 0.656 mm^{-1}. CCDC-797903.

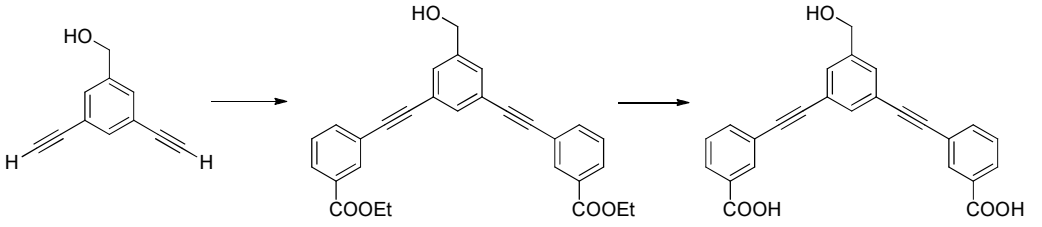
Crystal data for **MOP 3**: $C_{124}H_{96}Cu_4O_{28}$, M = 2288.17, blue prism, 0.30×0.20×0.10 mm^3, monoclinic, space group $C2/c$ (No. 15), a = 26.047(5), b = 18.101(3), c = 33.213(6) Å, β = 105.478(2)°, V = 15091(5) Å^3, Z = 4, D_c = 1.007 g/cm^3, F_{000} = 4720, MoKα radiation, λ = 0.71073 Å, T = 110(2)K, $2\theta_{max}$ = 52.0º, 56618 reflections collected, 14663 unique (R_{int} = 0.1011). Final *GooF* = 1.049, *R1* = 0.0940, *wR2* = 0.2035, *R* indices based on 6129 reflections with I I>2(I) (refinement on F^2), 494 parameters, 216 restraints. μ = 0.613 mm^{-1}. CCDC-797904.

Crystal data for **MOP 4**: $C_{169}H_{191}Cu_4N_4O_{29}$, M = 2996.42, blue prism, 0.12×0.08×0.07 mm^3, monoclinic, space group $P2_1/n$ (No. 14), a = 15.2237(9), b = 28.909(2), c = 36.303(2) Å, β = 100.644(1)°, V = 15701(2) Å^3, Z = 4, D_c = 1.268

g/cm^3, F_{000} = 6324, synchrotron radiation, λ = 0.41328 Å, T = 100(2)K, $2\theta_{max}$ = 30.0°, 211393 reflections collected, 31563 unique (R_{int} = 0.1063). Final *GooF* = 0.978, *R1* = 0.1196, *wR2* = 0.2281, *R* indices based on 16598 reflections with I I>2(I) (refinement on F^2), 1510 parameters, 155 restraints. μ = 0.326mm^{-1}. CCDC-797905.

3. Syntheses, Reactions, and Characterizations

3.1 Synthesis of 3,3'-((5-hydroxymethyl)-1,3phenylene)bis(ethyne-2,1-diyl))dibenzoic acid, H_2L



1 **2** **H_2L**

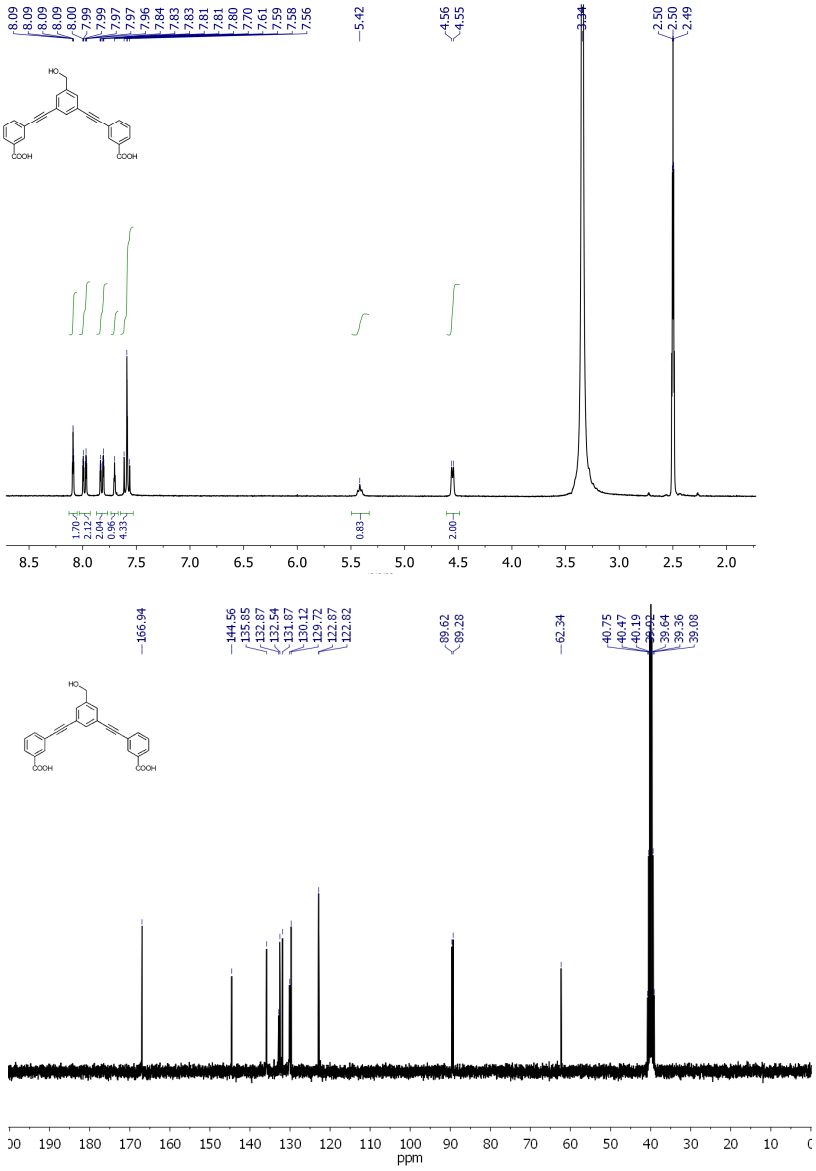
3.1.1 Synthesis of diethyl 3,3'-((hydroxyemthyl)-1,3,phenylene)bis(ethyne-2,1-diyl))dibenzoate,2.[1]

3,5-diethnylbenzyl alcohol (**1** 2.0 g, 12.8 mmol, synthesized according to literature[2]), $Pd(PPh_3)_4$ (300 mg, 0.26 mmol), and CuI (25.0 mg, 0.13 mmol) were placed in a 250 ml three necked round bottom flask equipped with a reflux condenser. Under nitrogen atmosphere and ice bath, 100 mL of degassed triethylamine was introduced in via cannula, and then ethyl 3-iodobenzoate (4.3 mL, 25.6 mmol) was syringed into above mixture. The system was then heated at 70°C for 12 h. Cooled to room temperature, precipitates were removed by filtration and the filtrate was evaporated to remove most of organic solvents, extracted with dichloromethane, washed with saturated $NaHCO_3$, water, brine, and then dried with $MgSO_4$. Filtered, and concentrated to dryness. The product was purified by column chromatography (hexanes/ethyl acetate, 4/1) to afford compound **2** (4.9 g, 85%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.12 (brs, 2H), 7.93 (d, 2H), 7.61 (d, 2H), 7.56 (brs, 1H), 7.46 (brs, 2H), 7.34 (t, 2H), 4.66 (brs, 2H), 4.32 (q, 4H), 3.56 (brs, 1H), 1.36(t, 6H).

*3.1.2 Synthesis of 3,3'-((5-hydroxymethyl)-1,3phenylene)bis(ethyne-2,1-diyl))dibenzoic acid, **H_2L***

10 mL of 2 mol/L NaOH was added to a solution of compound **2** (3.9 g, 8.6 mmol) in 100 mL THF/MeOH (v/v = 1/1), the resulting mixture was stirred overnight at room temperature. Most of organic solvents were removed by Rota vapor, the resulting

water solution was diluted to 50 mL, and concentrated HCl was added to adjust the PH value to 2. The white precipitate was collected, washed with water, and dried under vacuum to afford **H_2L** (2.8 g, 82%). ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.09 (t, 2H), 7.98 (dt, 2H), 7.82 (dt, 2H), 7.70 (t, 1H), 7.59 (brs, 2H), 7.58 (t, 2H), 5.42 (t, 1H), 4.55 (d, 2H) ppm; ^{13}C NMR (75 MHz, DMSO-d_6) δ 166.94, 144.56, 135.85, 132.87, 132.54, 131.87, 130.12, 130.06, 129.72, 122.87, 122.82, 89.61, 89.28, 62.34 ppm; *m/z* (ESI) 167.07 (100%), 209.11 (59%) and 395.13 (M^-, 73%). Elemental analysis (% calc/found: C 75.75/72.55, H 4.07/3.82). TGA was shown in **Fig. S2**.



3.2 Synthesis of MOP 1a

To a vial containing an N,N-diethylformamide (DEF, 2.0 mL) solution of **H_2L** (39 mg, 0.1 mmol) and $Cu(NO_3)_2$•$2.5H_2O$ (27 mg, 0.1 mmol) add 44 mg of 2,6-

dimethylpyridine (0.4 mmol). The resulting solution was layer with 2.0 mL of methanol, and the vial was sealed, allowed to stand at room temperature. 3 days later, Prismatic crystals were collected, washed with a little DEF and methanol, and then dried in air (Yield: 25 mg). PXRD of as-synthesized **MOP 1a** was shown in **Fig. S4**, TGA in **Fig. S3**, and IR in **Fig. S1**.

3.3 Synthesis of MOP 1b

To a vial containing an dimethyl sulfoxide (DMSO, 2.0 mL) solution of **H_2L** (39 mg, 0.1 mmol) and $Cu(NO_3)_2$•$2.5H_2O$ (27 mg, 0.1 mmol) add 132 mg of 2,6-dimethylpyridine (1.2 mmol). The resulting solution was layer with 2.0 mL of acetone, and the vial was sealed, allowed to stand at room temperature. 3 days later, prismatic crystals were collected, washed with a little DMSO and acetone, and then dried in air (Yield: 30 mg). PXRD of as synthesized **MOP 1b** was shown in **Fig. S5**, TGA in **Fig. S3**,and IR in **Fig. S1**.

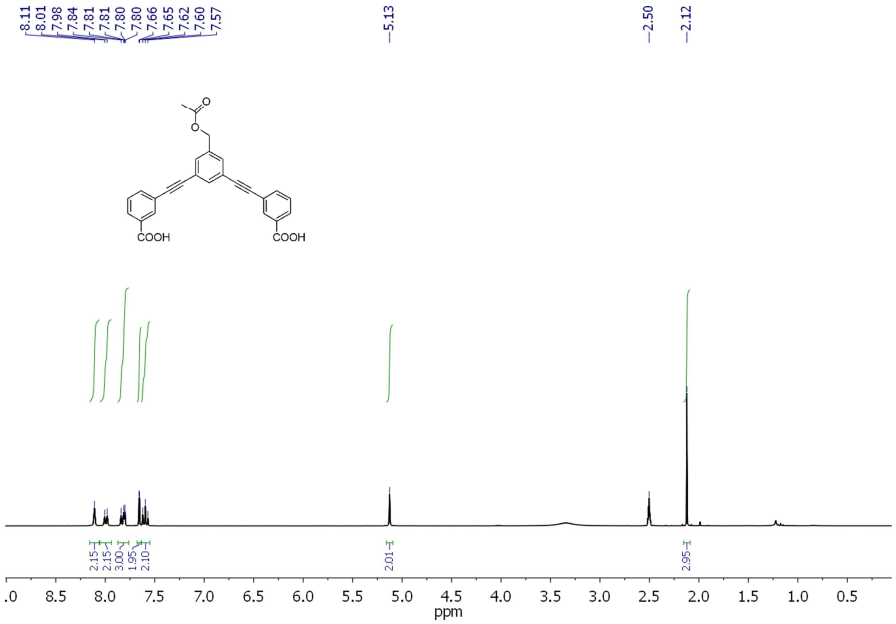
3.4 Activated MOP 1

MOP 1a or **MOP 1b** was soaked in methanol for 3 days (replaced with fresh methanol three times), and then dichloromethane for 3 days (replaced with fresh dichloromethane three times). After solvent was decanted, the solid was dried under dynamic vacuum at 100^0C for 12 h (color changed from green to blue). TGA of **activated MOP 1** was shown in **Fig. S2**.

3.5 Synthesis of MOP 2

To a solution of **as-activated MOP 1** (50 mg, MW: 1831.69, 0.027 mmol) and DMAP (4.0 mg, MW: 122, 0.033 mmol) in dried DMF (10 mL) add acetic anhydride (25 μL, MW: 102.09, 0.26 mmol), the resulting mixture was stirred at room temperature under N_2 overnight. DMF was removed under high vacuum at room temperature, 20 mL methanol was added; the resulting green suspension was centrifuged, and the supernatant was carefully decanted. Fresh methanol (20 mL) was added to the residue, stirred for a while, centrifugation and supernatant removal were repeated twice, The resulted green solid was dried under vacuum to produce 55 mg blue powder (~100% yield). 20 mg of above blue powder was dispersed in 10 mL of 1.0 mol/L HCl, sonicated 10 min, the resulted white suspension was collected with centrifuge, washed with water three times, dried over $Mg(SO_4)_2$, Filtered, and concentrated to dryness, The product was dried under vacuum (17 mg), and checked with ^{1}H-NMR. (300 MHz, DMSO-d_6): δ 8.11 (brt, 2H), 7.99 (dt, *J* = 7.8 *Hz*, 2H), 7.82

(dt, *J* = 7.8 *Hz*, 2H), 7.80 (t, *J* = 1.5 *Hz*, 1H), 7.66 (d, *J* = 1.5 *Hz*, 2H), 7.60 (t, *J* = 7.8 *Hz*, 2H), 5.13 (s, 2H), 2.12 (s, 3H) ppm.

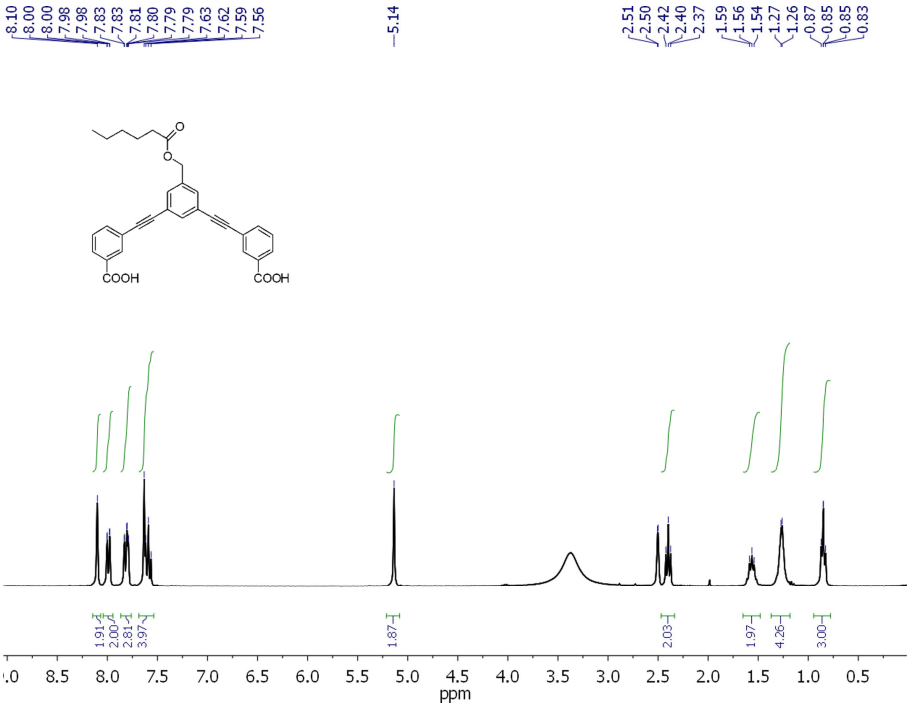


A 3.0 mL inner vial containing a solution of 20 mg above blue powder in 1.0 mL DEF was placed inside a 20 mL outer vial containing 3.0 mL methanol, the outer vial was then sealed. Green block crystals were formed and collected after the solution in the inner vial was colorless. PXRD of as-synthesized **MOP 2** was shown in **Fig. S6**, TGA in **Fig. S3**, and IR in **Fig. S1**.

3.6 Synthesis of MOP 3

To a solution of **as-activated MOP 1** (50 mg, MW: 1831.69, 0.027 mmol) and DMAP (4.0 mg, MW: 122, 0.033 mmol) in dried DMF (10 mL) add hexanoic anhydride (60 μL, MW: 214.3, 0.26 mmol), the resulting mixture was stirred at room temperature under N_2 overnight. Most of DMF was removed under high vacuum at room temperature, 20 mL methanol was added; the resulting green suspension was centrifuged, and the supernatant was carefully decanted. Fresh methanol (20 mL) was added to the residue, stirred for a while, centrifugation and supernatant removal were repeated twice, the resulted green solid was dried under vacuum to produce 61 mg blue powder (~100% yield). 20 mg of above blue powder was dispersed in 10 mL of 1.0 mol/L HCl, sonicated 10 min, the resulted white suspension was collected with centrifuge, washed with water three times, dried over $Mg(SO_4)_2$, Filtered, and concentrated to dryness, The product was dried under vacuum (17 mg), and checked with ^{1}H-NMR (300 MHz, DMSO-d_6): δ 8.10 (brt, 2H), 7.99 (dt, *J* = 7.8 *Hz*, 2H), 7.82

(dt, *J* = 7.8 *Hz*, 2H), 7.79 (t, *J* = 1.5 *Hz*, 1H), 7.63 (brd, 2H), 7.59 (t, *J* = 7.8 *Hz*, 2H), 5.14 (s, 2H), 2.40 (t, *J* = 7.2 *Hz*, 2H), 1.56 (m, 2H), 1.27 (m, 4H) 0.85 (t, *J* = 8.7 *Hz*, 3H) ppm.

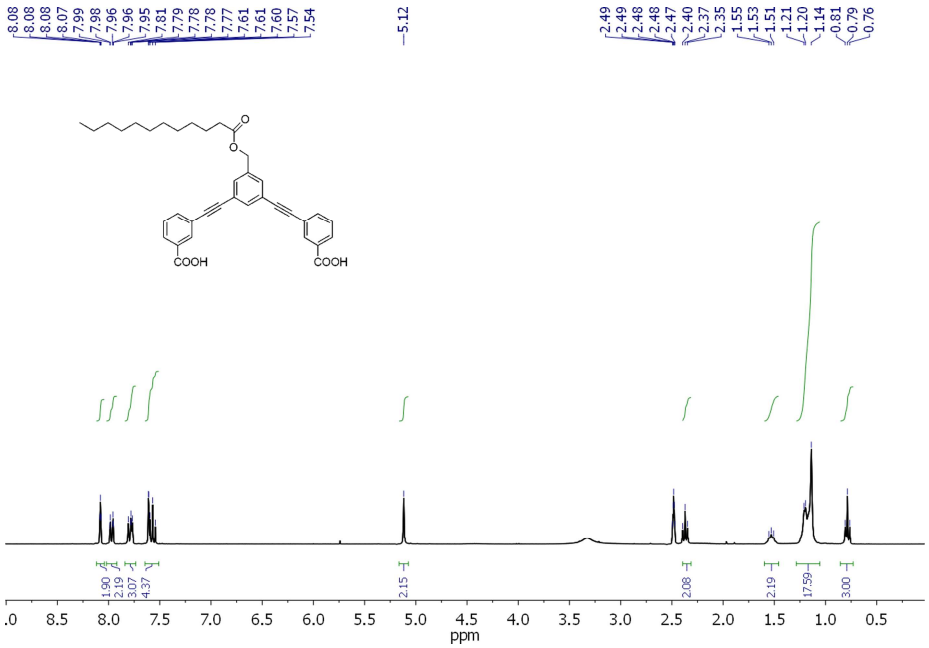


A 3.0 mL inner vial containing a solution of 20 mg above blue powder in 1.0 mL DEF was placed inside a 20 mL outer vial containing 3.0 mL methanol, the outer vial was then sealed. Prismatic crystals were formed and collected after the solution in the inner vial was colorless. PXRD of as-synthesized **MOP 3** was shown in **Fig. S7**, TGA in **Fig. S3**, and IR in **Fig. S1**.

3.7 Synthesis of MOP 4

To a solution of **as-activated MOP 1** (50 mg, MW: 1831.69, 0.027 mmol) and DMAP (4.0 mg, MW: 122, 0.033 mmol) in dried DMF (10 mL) add Lauric anhydride (100 mg, MW: 382.63, 0.26 mmol), the resulting mixture was stirred at room temperature under N_2 overnight. Most of DMF was removed under high vacuum at room temperature, 20 mL methanol was added; the resulting green suspension was centrifuged, and the supernatant was carefully decanted. Fresh methanol (20 mL) was added to the residue, stirred for a while, centrifugation and supernatant removal were repeated twice, and the resulted green solid was dried under vacuum to produce 68 mg blue powder (~100% yield). 20 mg of above blue powder was dispersed in 10 mL of 1.0 mol/L HCl, sonicated 10min, the resulted white suspension was collected with centrifuge, washed with water three times, dried over $Mg(SO_4)_2$, Filtered, and

concentrated to dryness, The product was dried under vacuum (18 mg), and checked with ^{1}H-NMR (300 MHz, DMSO-d_6): δ 8.10 (t, *J* = 1.5 *Hz*, 2H), 7.99 (dt, *J* = 7.8 and 1.5 *Hz*, 2H), 7.82 (dt, *J* = 7.8 and 1.5 *Hz*, 2H), 7.79 (t, *J* = 1.5 *Hz*, 1H), 7.63 (d, *J* = 1.5 *Hz*, 2H), 7.59 (t, *J* = 7.8 *Hz*, 2H), 5.14 (s, 2H), 2.39 (t, *J* = 7.2 *Hz*, 2H), 1.55 (m, 2H), 1.23-1.16 (m, 18H) 0.80 (t, *J* = 7.8 *Hz*, 3H) ppm.



A 3.0 mL inner vial containing a solution of 20 mg above blue powder in 1.0 mL DEF was placed inside a 20 mL outer vial containing 3.0 mL methanol, the outer vial was then sealed. Green block crystals were formed and collected after the solution in the inner vial was colorless. PXRD of as-synthesized **MOP 4** was shown in **Fig. S8**, TGA in **Fig. S3**,and IR in **Fig. S1**.

4. Infrared spectra (IR)

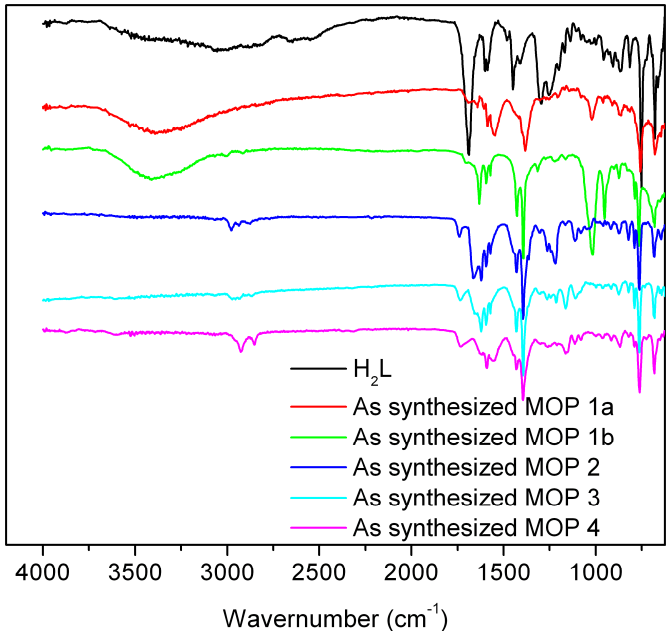


Fig. S1 IR spectra of H_2L and as-synthesized MOP 1a, MOP 1b, MOP 2, MOP 3, and MOP 4.

5. Thermogravimetric Analysis (TGA)

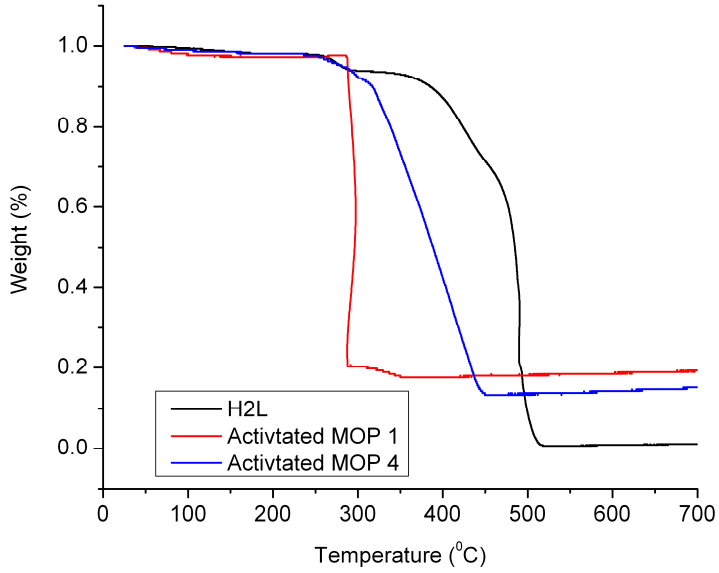


Fig. S2 TGA curves of H_2L, activated MOP 1, and MOP 4.

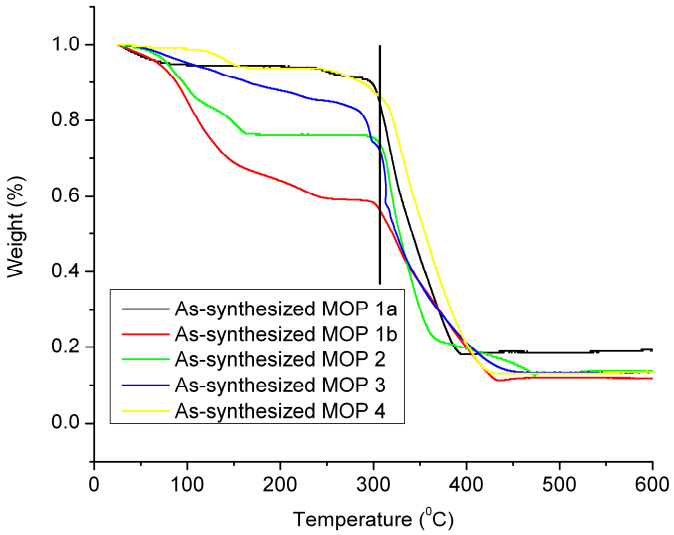


Fig. S3 TGA curves of as-synthesized MOP 1a, MOP 1b, MOP 2, MOP 3, and MOP 4.

6. Powder X-ray Diffraction (PXRD)

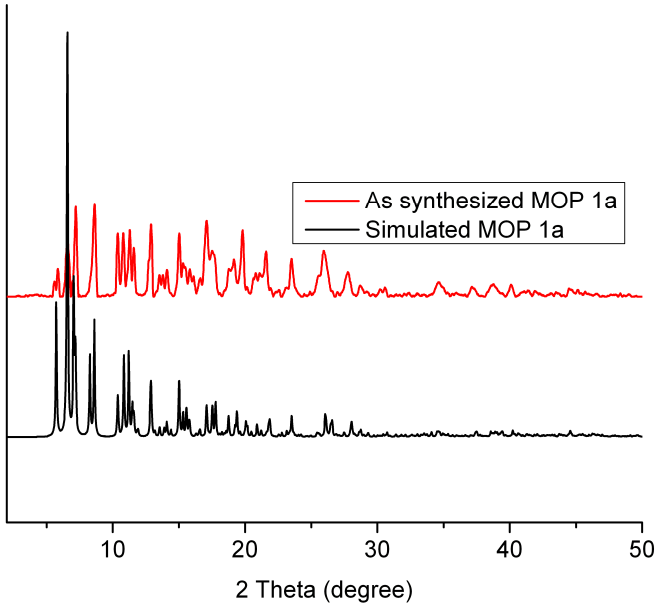


Fig. S4 Powder X-ray diffraction (PXRD) patterns of MOP 1a: pattern simulated from single crystal structure in black; observed pattern for the prepared sample in red.

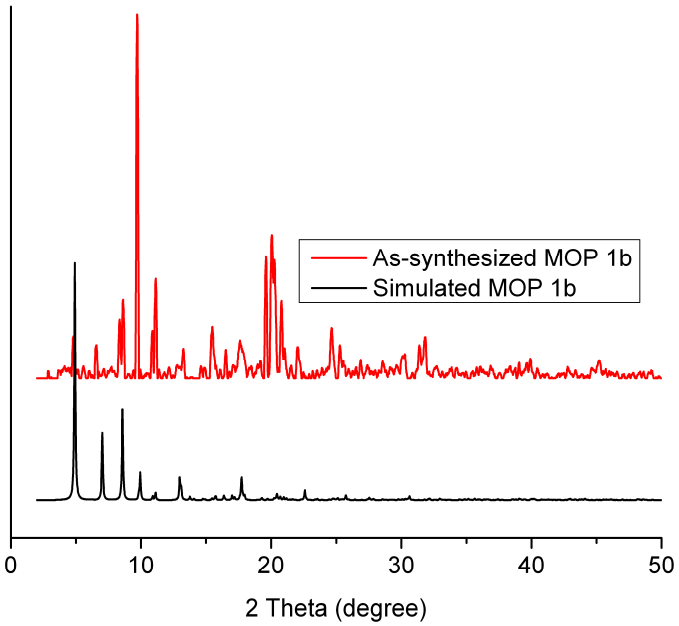


Fig. S5 Powder X-ray diffraction (PXRD) patterns of MOP 1b: pattern simulated from single crystal structure in black; observed pattern for the prepared sample in red.

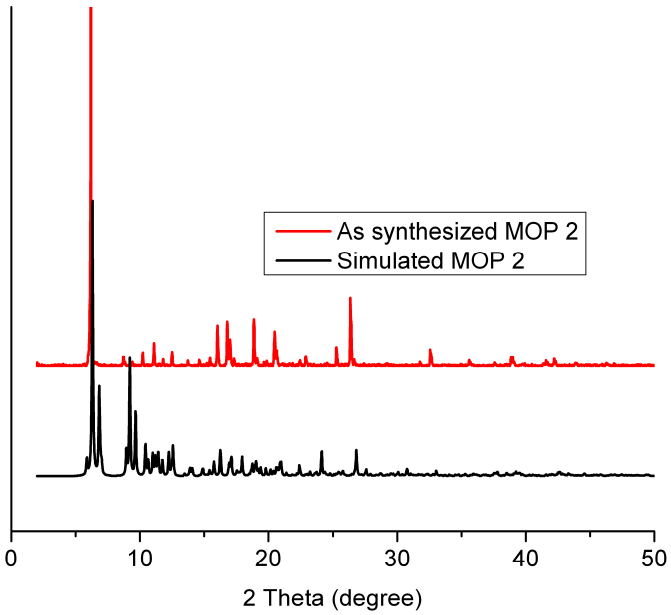


Fig. S6 Powder X-ray diffraction (PXRD) patterns of MOP 2: pattern simulated from single crystal structure in black; observed pattern for the prepared sample in red.

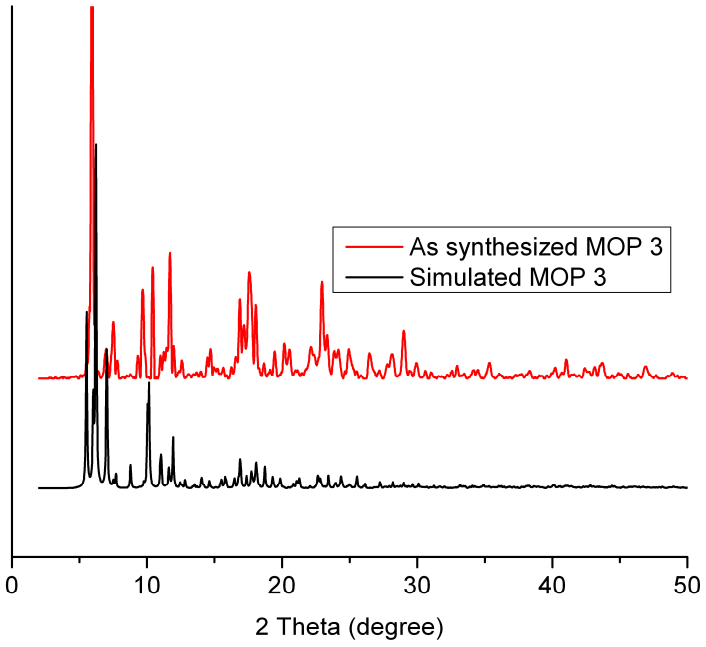


Fig. S7 Powder X-ray diffraction (PXRD) patterns of MOP 3: pattern simulated from single crystal structure in black; observed pattern for the prepared sample in red.

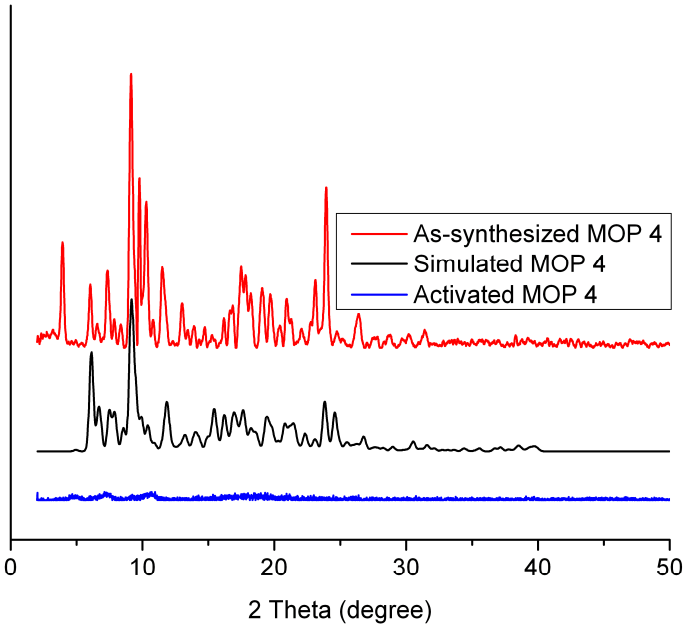


Fig. S8 Powder X-ray diffraction (PXRD) patterns of MOP 4: pattern simulated from single crystal structure in black; observed pattern for the prepared sample in red; and observed pattern for activated sample in blue.

7. Gas Adsorption

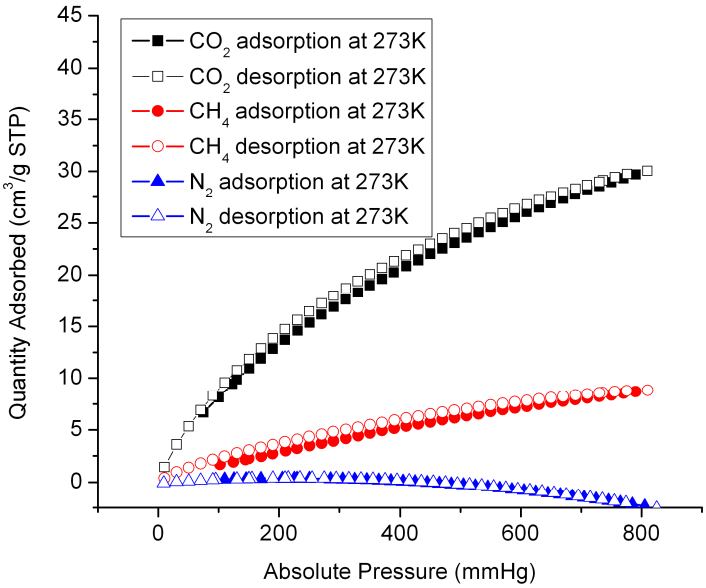


Fig. S9 CO_2, CH_4, and N_2 sorption isotherms of activated MOP 1 at 273K (215mg sample used for measurement).

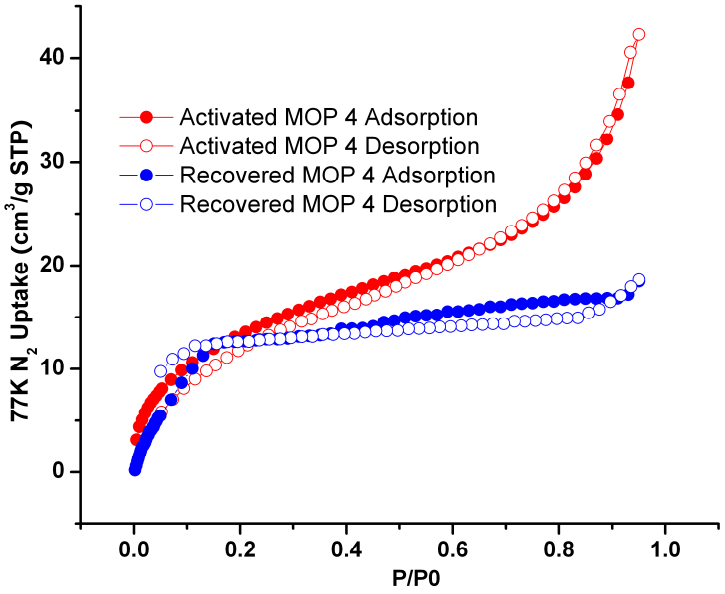


Fig. S10 N_2 sorption isotherms of activated MOP 4 and recovered MOP 4 at 77K.

8. Absorption spectra

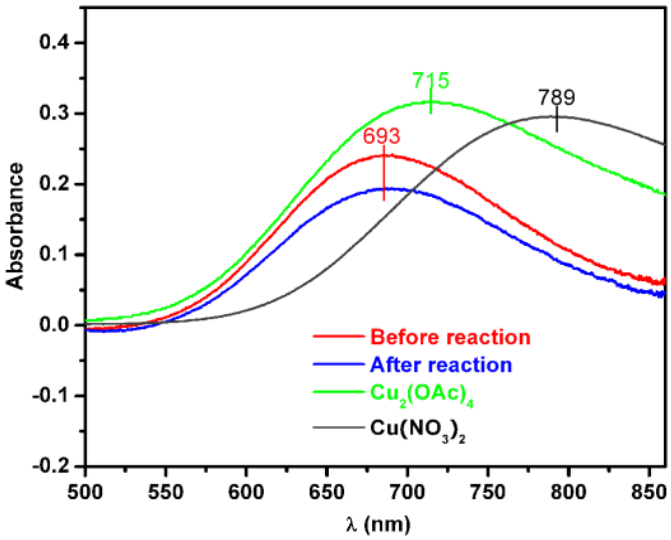
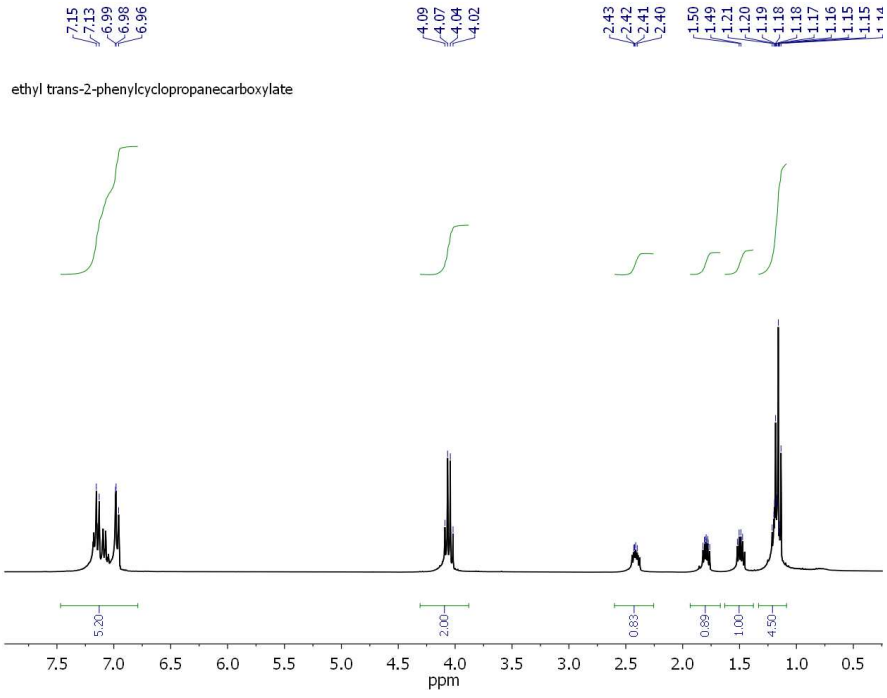
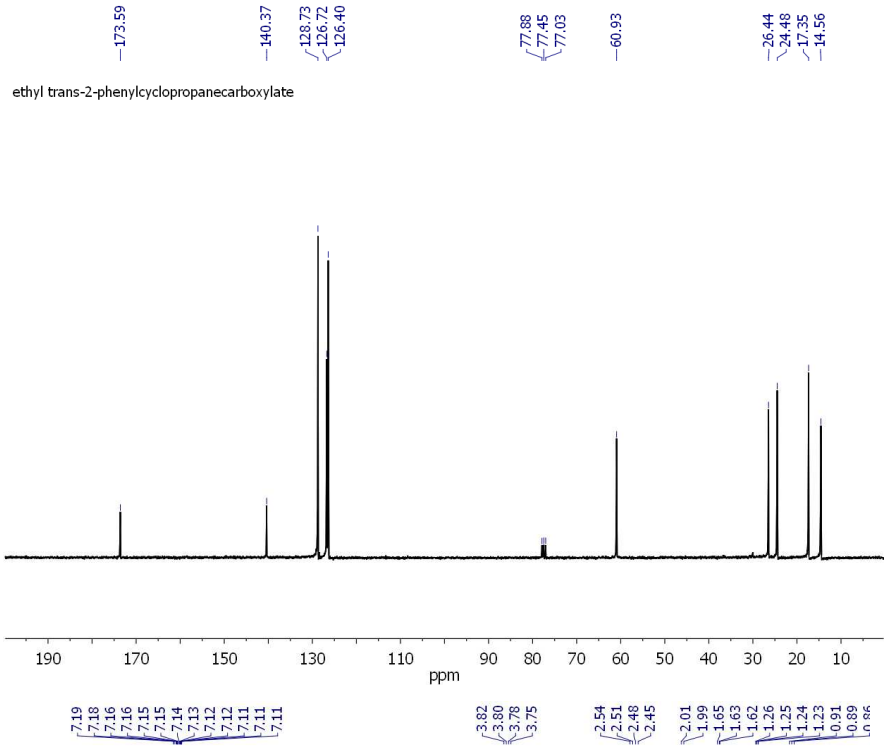


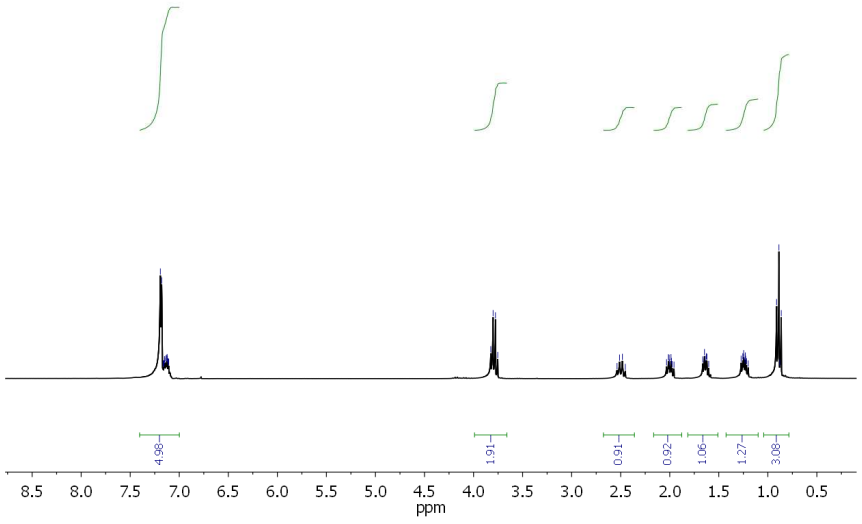
Fig. S11 Absorption spectra of MOP 4 before (red) and after (blue) the catalytic reaction, copper acetate (green), as well as copper nitrate (black).

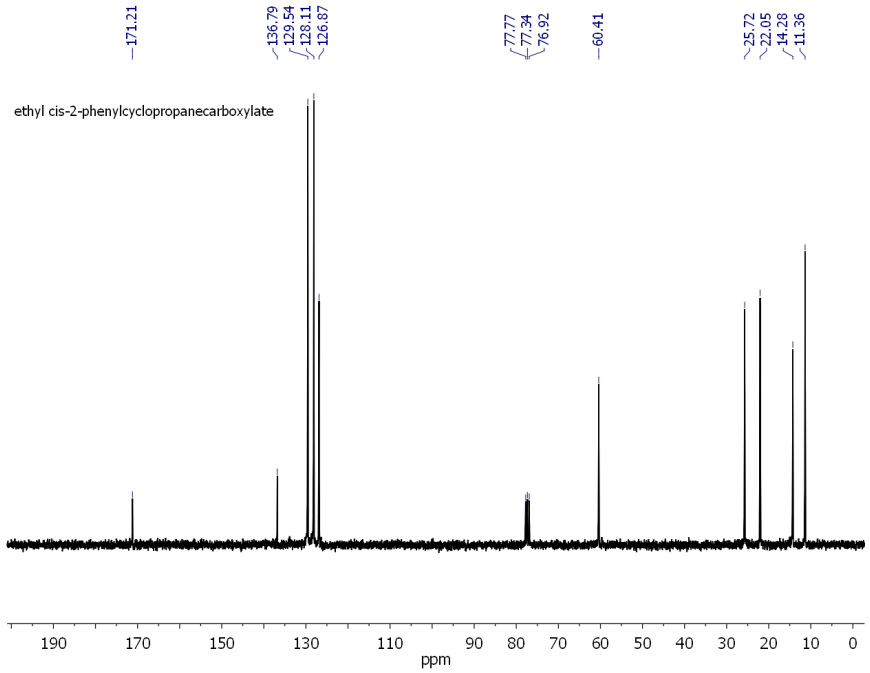
9. HPLC data

Ethyl trans-2-phenylcyclopropanecarboxylate and its cis isomer were separated from the reaction of styrene and EDA with MOP-4 as catalyst. Their NMR data were in good agreement with Literatures'.




173.59
140.37
128.73
126.72
126.40
77.88
77.45
77.03
60.93
26.44
24.48
17.35
14.56
ethyl trans-2-phenylcyclopropanecarboxylate
190
170
150
130
110
90
80
70
60
50
40
30
20
10
ppm
7.19
7.18
7.16
7.16
7.15
7.15
7.14
7.13
7.12
7.12
7.11
7.11
7.11
3.82
3.80
3.78
3.75
2.54
2.51
2.48
2.45
2.01
1.99
1.65
1.63
1.62
1.26
1.25
1.24
1.23
0.91
0.89

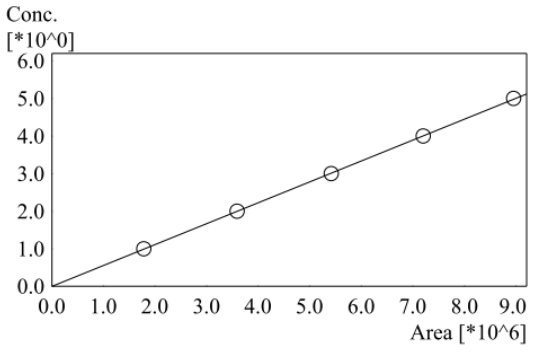

ethyl cis-2-phenylcyclopropanecarboxylate
4.98
1.91
0.91
0.92
1.06
1.27
3.08
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm



9.1 Trans and Cis calibration

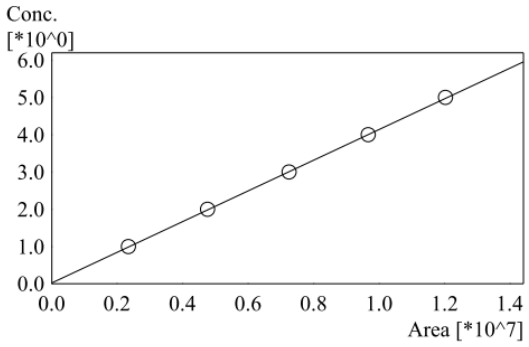
Calibration solution: 0.5 mg ethyl trans-2-phenylcyclopropanecarboxylate and 0.5 mg ethyl cis-2-phenylcyclopropanecarboxylate was dissolved in 1.0 mL methanol.

ID# : 1
Name : Cis
Quantitative Method : External Standard
Function : f(x)=5.5734e-007*x-0.00404801
Rr1=0.9999672 Rr2=0.9999344
MeanRF:5.56899e-007 RFSD:2.699e-009 RFRSD:0.484648
FitType : Linear
ZeroThrough : Not Through
Weighted Regression : None
Detector Name : PDA



#	Conc. (Ratio)	MeanArea	Area
1	1.000	1784938.6	1788206
			1781140
			1785470
2	2.000	3593098.7	3604515
			3600553
			3574228
3	3.000	5419739.0	5431052
			5403658
			5424507
4	4.000	7200023.7	7196682
			7200207
			7203182
5	5.000	8952073.4	8956711
			8959308
			8940201

ID# : 2
Name : Trans
Quantitative Method : External Standard
Function : f(x)=4.1217e-007*x+0.0271351
Rr1=0.9999590 Rr2=0.9999181
MeanRF:4.17703e-007 RFSD:5.52846e-009 RFRSD:1.32354
FitType : Linear
ZeroThrough : Not Through
Weighted Regression : None
Detector Name : PDA



#	Conc. (Ratio)	MeanArea	Area
1	1.000	2349585.3	2364642
			2340547
			2343567
2	2.000	4763252.0	4812180
			4801372
			4676204
3	3.000	7255235.8	7246887
			7251092
			7267729
4	4.000	9668724.4	9652383
			9647040
			9706750
5	5.000	12026768.4	12068225
			12040548
			11971532

9.2 First run

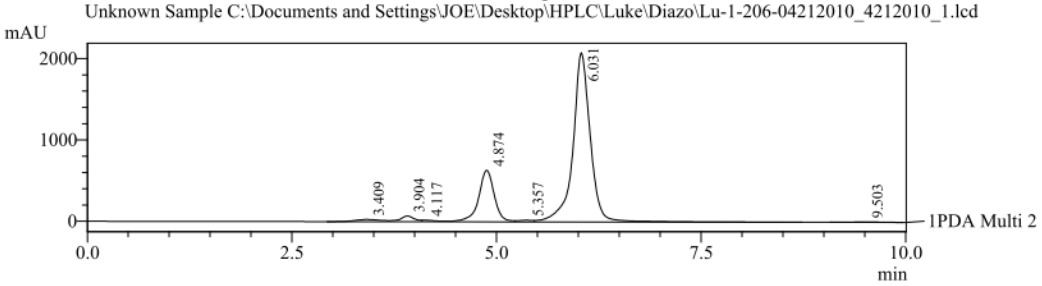
0.3 mL EDA (2.4 mmol) was dissolved in 5.0 mL dichloromethane, and was added in 10 h into a stirred solution of 0.5 mL styrene, and 15 mg activated MOP 4 (0.024 mmol open copper sites) in 5.0 mL dichloromethane at room temperature, stirred for another 10 h. 10 mL methanol was added, MOP 4 was precipitated, collected, dried, and used for next run. The filtrate was diluted to 250 mL with methanol, and then used for HPLC analysis.

Sample Information

Acquired by	: Admin
Sample Name	: Unknown Sample
Sample ID	: UNK-0001
Tray#	: 1
Vail#	: 14
Injection Volume	: 5 uL
Data Filename	: Lu-1-206-04212010_4212010_1.lcd
Method Filename	: Shodex ORpak 60%MeOH 0.6mL.lcm
Report Filename	: Default.lcr
Date Acquired	: 4/23/2010 2:55:56 PM

Chromatogram

Unknown Sample C:\Documents and Settings\JOE\Desktop\HPLC\Luke\Diazo\Lu-1-206-04212010_4212010_1.lcd



1 PDA Multi 2 / 217nm 4nm

Quantitative Results C:\Documents and Settings\JOE\Desktop\HPLC\Luke\Diazo\Lu-1-206-04212010_4212010_1.lcd

PDA

ID#	Name	Ret. Time	Area	Conc.
1	Cis	4.874	8447513	4.704
2	Trans	6.031	30785539	12.716

9.3 Second run

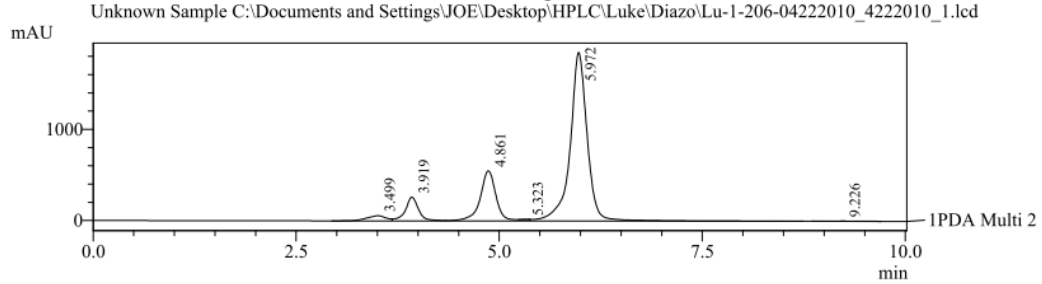
Same procedure as first run instead of 4.0 mg MOP-4 recovered from first run used.

Sample Information

Acquired by	: Admin
Sample Name	: Unknown Sample
Sample ID	: UNK-0001
Tray#	: 1
Vail#	: 15
Injection Volume	: 5 uL
Data Filename	: Lu-1-206-04222010_4222010_1.lcd
Method Filename	: Shodex ORpak 60%MeOH 0.6mL.lcm
Report Filename	: Default.lcr
Date Acquired	: 4/23/2010 3:49:45 PM

Chromatogram

Unknown Sample C:\Documents and Settings\JOE\Desktop\HPLC\Luke\Diazo\Lu-1-206-04222010_4222010_1.lcd



1 PDA Multi 2 / 217nm 4nm

Quantitative Results C:\Documents and Settings\JOE\Desktop\HPLC\Luke\Diazo\Lu-1-206-04222010_4222010_1.lcd

PDA

ID#	Name	Ret. Time	Area	Conc.
1	Cis	4.861	7550692	4.204
2	Trans	5.972	27224140	11.248

10. References

[1] G. M. Sheldrick, *SHELXTL*, Version 6.14, Structure Determination Software Suite, Bruker AXS, Madison, WI, **2003**.
[2] A. L. Spek, *PLATON*, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands, **1998**.
[3] E. Reisner, S. J. Lippard, *Eur. J. Org. Chem.* **2008**, *2008*, 156-163.
[4] K. Fukushima, A. J. Vandenbos, T. Fujiwara, *Chem. Mater.* **2007**, *19*, 644-646.