Supporting Information

An Isolated Doughnut-like Molybdenum(V) Cobalto-phosphate Cluster Displaying Excellent Photocatalytic Performance of Carbon Dioxide Conversion

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1. Experimental section

1.1 Materials and analytical methods

All the starting chemicals were commercially available and used with no further purification. PXRD analysis (5−50°) of the compound was performed on Bruker D8X diffractometer with the monochromatized Cu-Kα (λ = 0.15418 nm) radiation. IR spectra (4000−400 cm^{-1}) were recorded by a Nicolet Impact 410 FTIR spectrometer using pressed KBr pellets. TG measurement (25−1000 °C) was carried out on a Diamond thermogravimetric analyzer in flowing N_2 atmosphere, with a heating rate of 10 °C min^{-1}. The UV-Vis diffuse reflectance spectrum (λ = 200−800 nm) was investigated on SHIMADZU UV-2600 spectrophotometer with $BaSO_4$ as reference. Mott-Schottky plots were measured by a CHI 440 electrochemical quartz crystal microbalance that was connected to a Digital-586 personal computer.

1.2 Preparation of [Co@{$Co_{16}Mo_{16}$}]

$Co(CH_3COO)_2·4H_2O$ (0.5 mmol, 0.1245 g), MoO_3 (0.5 mmol, 0.0719 g) and 4-phenylpyridine (0.4 mmol, 0.066 g) were dissolved in a mixed solution of 1 mL tetrabutylammonium hydroxide, 4mL distilled water and 4 mL ethanol. The mixture was magnetic stirred for 30 min at room temperature, adjust the pH value of the mixture to about 3.5 with 85% H_3PO_4, and then continue to stir 120 min. The suspension was sealed into a stainless-steel autoclave with 25 mL Teflon-lined and maintained the temperature at 180 ℃ for 5 days. After falling to room temperature, yellow-brown diamond crystals were obtained by washing and filtering with distilled water. The yield of **[Co@{$Co_{16}Mo_{16}$}]** is 32% (based on Mo). Elemental analysis result (%): C,4.07; H,2.12; N,0.45. (Calcd (%):C, 3.95; H,2.17; N,0.42).

1.3X-ray crystallography

Single-crystal XRD data of the compound were collected at 296 K from a Bruker Apex II CCD with Mo-Kα radiation (λ = 0.71073 Å). The crystal structure was resolved by using direct methods and then refined via full-matrix least-squares on F^2 by means of the SHELXL-2018/3 program package. Some water molecules were removed by

SQUEEZE in the refinement, but accurately confirmed by TG and elemental analyses. A summary of the crystallographic data for the compound is provided in Table S1. CCDC-2157140 contains the supplementary crystallographic data for this paper.

1.4 Photocatalytic CO_2 reduction experiments

The photocatalytic performance of **[Co@{$Co_{16}Mo_{16}$}]** was evaluated by applying it to the photocatalytic reduction of CO_2 (CEL-SPH2N-S9, AULTT, China). The experiments were carried out in a 100 mL Pyrex flask. A 300 W xenon arc lamp (CEL-PF300-T8, AULTT, China) (photocurrent: 14.5A) was employed as a visible-light source through a UV-cutoff filter with a wavelength greater than 420 nm, which was installed 10 cm away from the reaction solution. In the system of CO_2 photocatalytic reduction, we put photocatalyst (5mg) into a mixed solvent of triethanolamine (TEOA, as a sacrificial base) and acetonitrile (1:4 v/v, 50 mL), and used $[Ru(bpy)_3]Cl_2$•$6H_2O$ (11.3mg) as photosensitizer. The products were analyzed by performing gas chromatography (GC7920-TF2Z, AULTT, China).

1.5 Electrochemical measurements

The Mott–Schottky spots were carried out at ambient environment via using the electrochemical workstation (CHI 760e) in a standard three-electrode system: The carbon cloth (CC, 1 cm×1 cm) modified with catalyst samples, carbon rod and Ag/AgCl were used as the working electrode, counter electrode, and the reference electrode, respectively. The catalyst of 5 mg was grinded to powder and then dispersed in 1 mL of 0.5% Nafion solvent by ultrasonication to form a homogeneous ink. Subsequently, 200 μL of the ink were deposited onto the carbon cloth, and dried in room temperature for Mott-Schottky spots measurements. The Mott-Schottky plots were measured over an alternating current (AC) frequency of 1000 Hz, 1500 Hz and 2000 Hz, and three electrodes were immersed in the 0.2 M Na_2SO_4 aqueous solution.

Table S1 Crystal unit cell parameters for **[Co@{$Co_{16}Mo_{16}$}]2D**.

[Co@{$Co_{16}Mo_{16}$}]2D	**Crystal system, space group**	**Unit cell dimensions**	**Volume**
[$(Mo_2O_4)_8(HPO_4)_{14}(PO_4)_{10}${ $Co_{19}Na_4(H_2O)_{34}$}]·$14H_2O$	Monoclinic $P2_1/n$	a=15.63 Å b=17.28 Å c=29.87 Å	7955 $Å^3$

Table S2 Crystallographic data of **[Co@{$Co_{16}Mo_{16}$}]**.

Compound	**[Co@{$Co_{16}Mo_{16}$}]**
Formula	C_{22} H_{145} Co_{17} Mo_{16} N_2 O_{183} P_{25}
Formula weight	6677.49
Crystal system	Monoclinic
Space group	*C*2/*c*
a (Å)	a = 33.455(12)
b (Å)	b = 20.185(12)
c (Å)	c = 30.076(13)
α (°)	90°
β (°)	100.038(7)°
γ (°)	90°
V ($Å^3$)	19999(16)
Temperature (K)	296(2)
Z	4
D_c (mg/m^3)	2.218 Mg/m^3
Absorption coefficient (mm^{-1})	2.655 mm^{-1}
F(000)	13044
Crystal size(mm)	0.14x0.12x 0.11mm^3
Limiting indices	-39<=h<=39 -24<=k<=24 -35<=l<=35
Reflections collected	69944
Independent reflections	17629 [R(int) = 0.1424]
Completeness	99.80%
Data/ restraints/ parameters	17629 / 298 / 1246
Goodness on F^2	1.007
R_1[a], wR_2[b] [I>2sigma(I)]	R1 = 0.0625, wR2 = 0.1355
R_1, wR_2 (all data)	R1 = 0.1246, wR2 = 0.1508

[a]$R_1 = \Sigma||F_o|-|F_c||/\Sigma|F_o|$; [b]$wR_2 = \Sigma[w(F_o^2-F_c^2)^2]/\Sigma[w(F_o^2)^2]^{1/2}$

2. Structural information

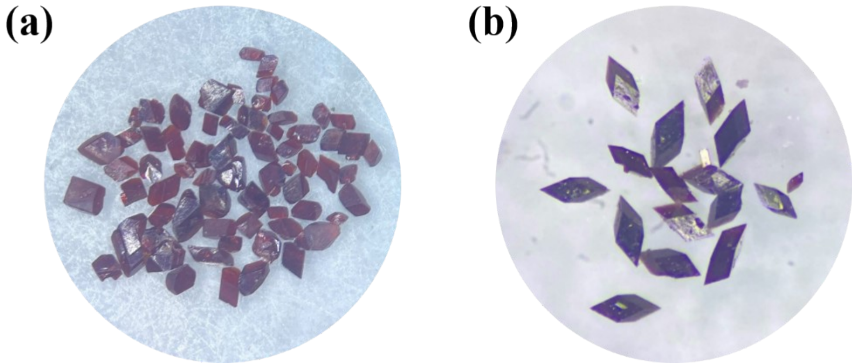

Fig. S1 (a)The image of **[Co@{$Co_{16}Mo_{16}$}]** crystal under optical microscope. (b) The image of **[Co@{$Co_{16}Mo_{16}$}]2D** crystal under optical microscope.

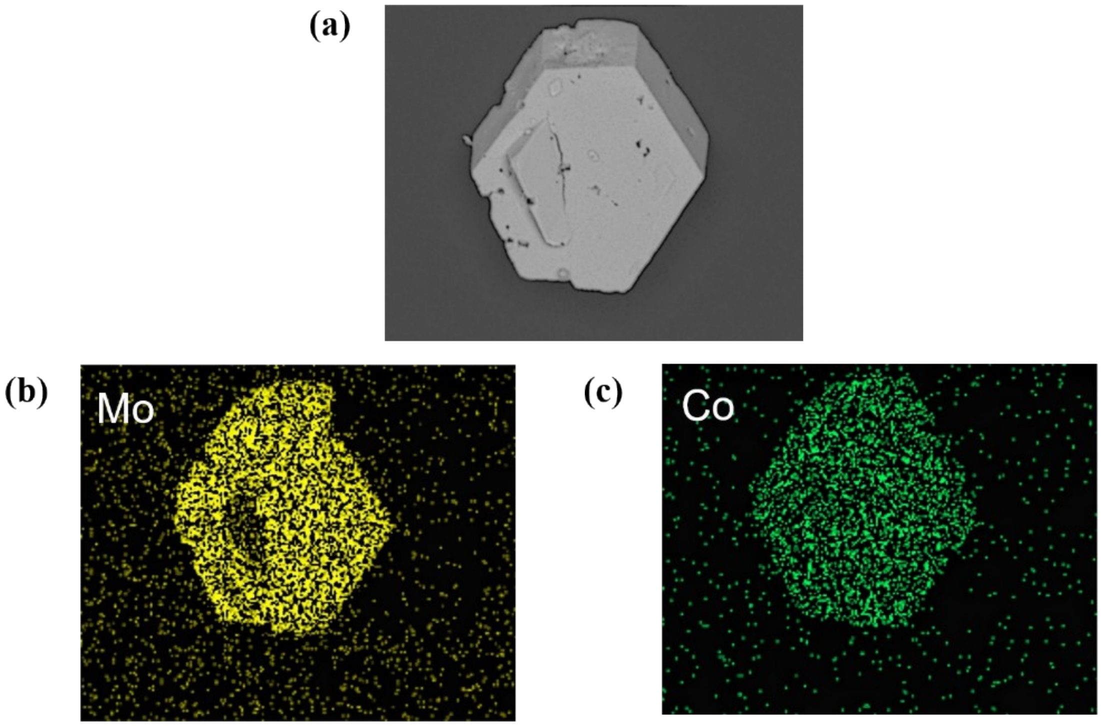

Fig. S2 (a)SEM pattern and (b, c) the mapping of Mo, Co elements of **[Co@{$Co_{16}Mo_{16}$}]**.

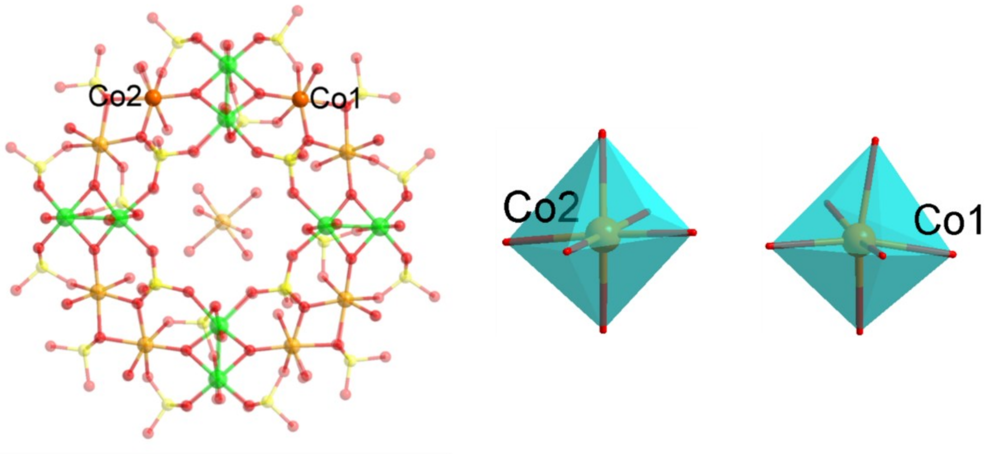

Fig S3. Coordination modes of two kinds of Co^{II} in **$\{Co_{16}Mo_{16}\}$**.

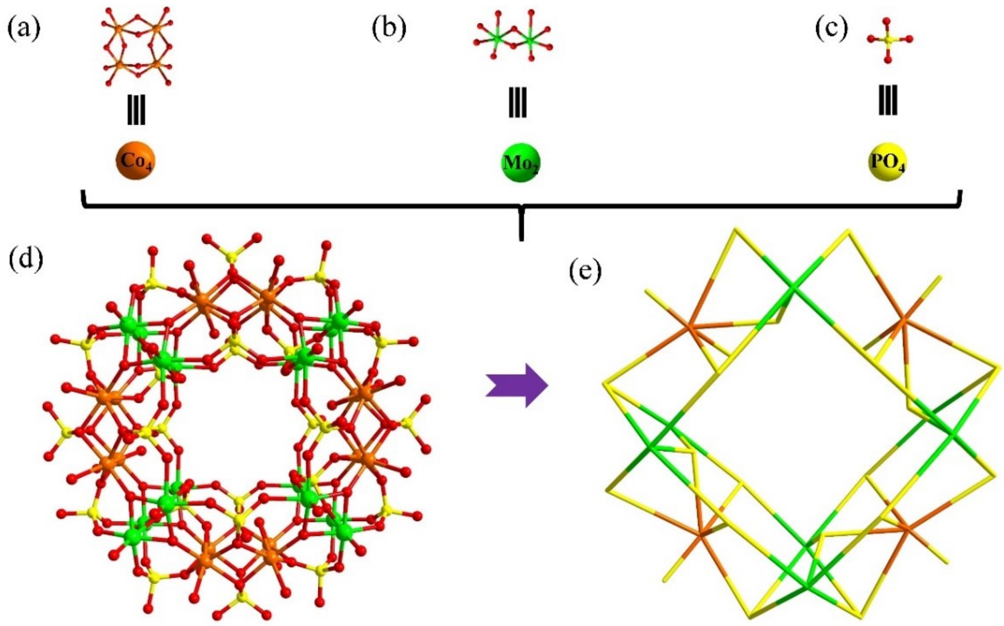

Fig. S4 (a)The simplified schematic diagram of the basic unit of $\{Co_4\}$ tetramer; (b)The simplified schematic diagram of the basic unit of $\{Mo_2\}$. (c) The simplified schematic diagram of the basic unit of Phosphate (PO_4). (d)The **$\{Co_{16}Mo_{16}\}$** ball and stick diagram. (e)The Simplified skeleton diagram of **$\{Co_{16}Mo_{16}\}$.**

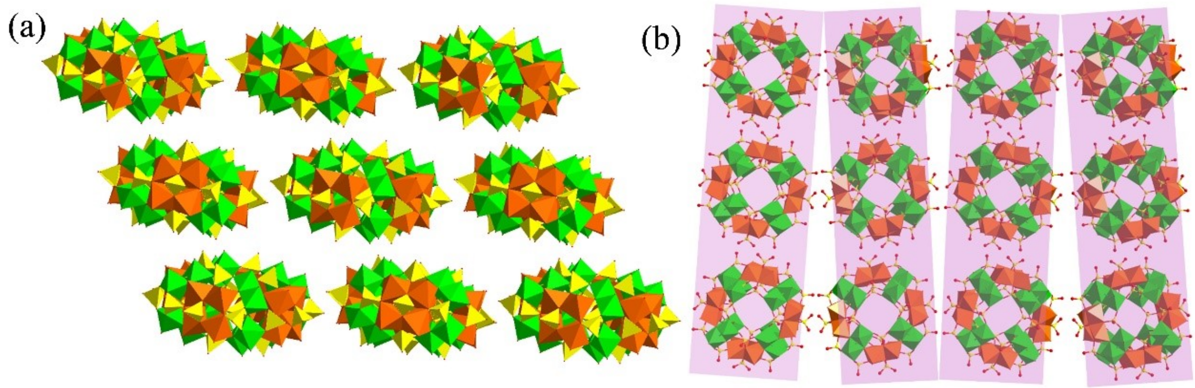

Fig. S5 (a) Side view (b) Plan view of **[Co@{$Co_{16}Mo_{16}$}]**. Free water, free ligands and hydrogen atoms are omitted for clarity.

3. Characterization information

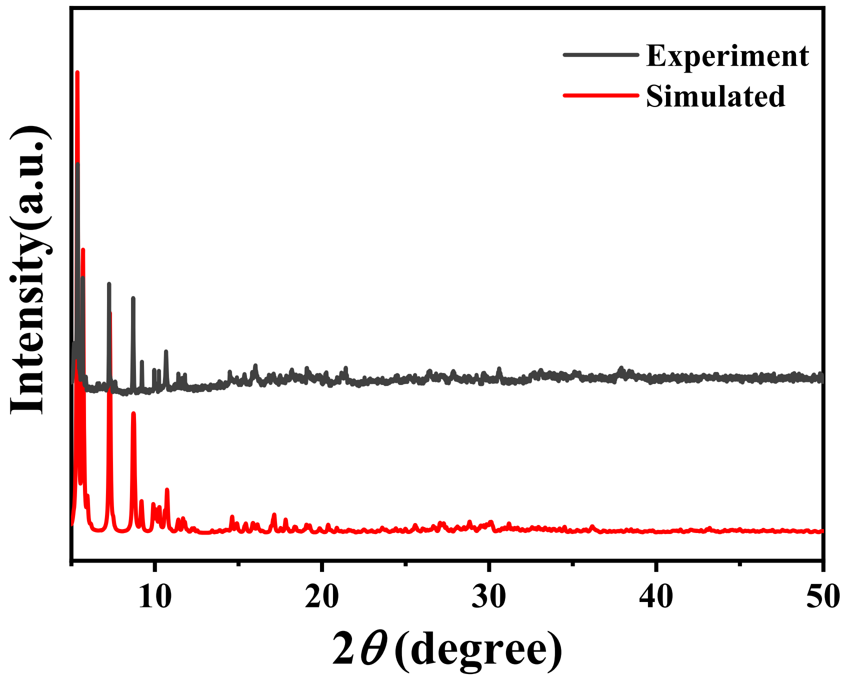

Fig. S6 Experimental and simulated PXRD patterns of **[Co@{$Co_{16}Mo_{16}$}]**.

PXRD curves were explored in order to evaluate the purity of **[Co@{$Co_{16}Mo_{16}$}]** (as shown in Fig. S6). The results of experiments for **[Co@{$Co_{16}Mo_{16}$}]** are the same as the fitting values, suggesting that products in this work are consistent with the pure materials.

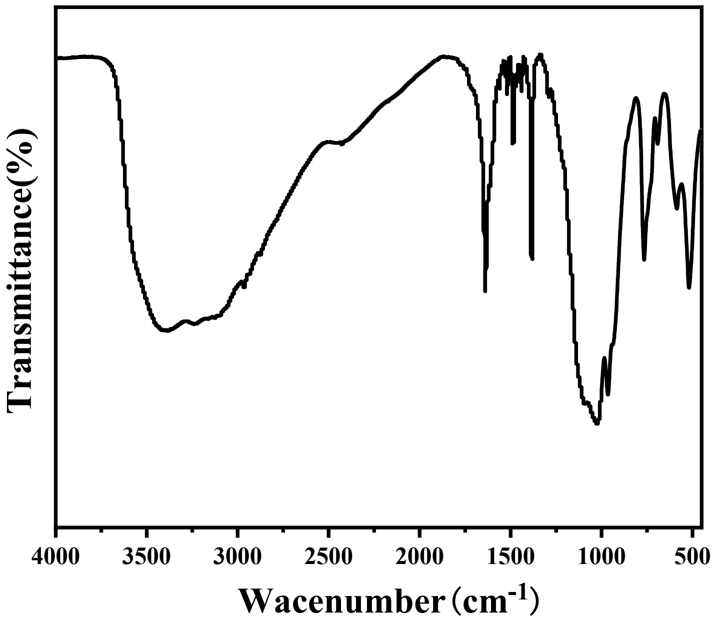

Fig. S7 The IR spectrum of **[Co@{$Co_{16}Mo_{16}$}]**.

The infrared spectra of **[Co@{$Co_{16}Mo_{16}$}]** is shown in Fig. S7. The absorption peak at 617-731 cm^{-1} is attributed to the ν(Mo-O-Mo) characteristic vibration peak. The absorption peak at 941-969 cm^{-1} is attributed to the vibration peak of the ν(Mo=O) bond, and 1012-1126 cm^{-1} is attributed to the vibration of the ν(P-O) bond. The absorption peak at 1443-1634 cm^{-1} is attributed to the stretching vibration peaks of the C-C bond and C-N bond on the ligand. The absorption peak at 3100-3421 cm^{-1} is attributed to the O–H bond stretching vibration from H_2O [1-3].

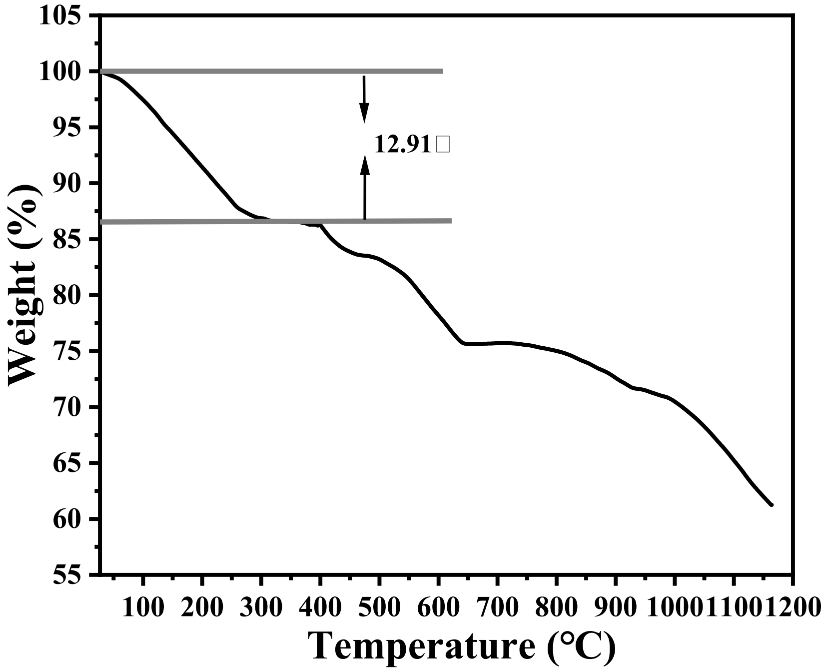

Fig. S8 The TG curve of **[Co@{$Co_{16}Mo_{16}$}]**.

The TGA curve of **[Co@{Co$_{16}$Mo$_{16}$}]** (as shown in Fig. S8) measured under synthetic air atmosphere from room temperature to 1200 °C at the hating rate of 10 °C min^{-1}. A continuous weight loss of 12.91 % (theoretical 12.67%) step from 25 to 320 °C corresponds to the loss of all lattice water and free water molecules.

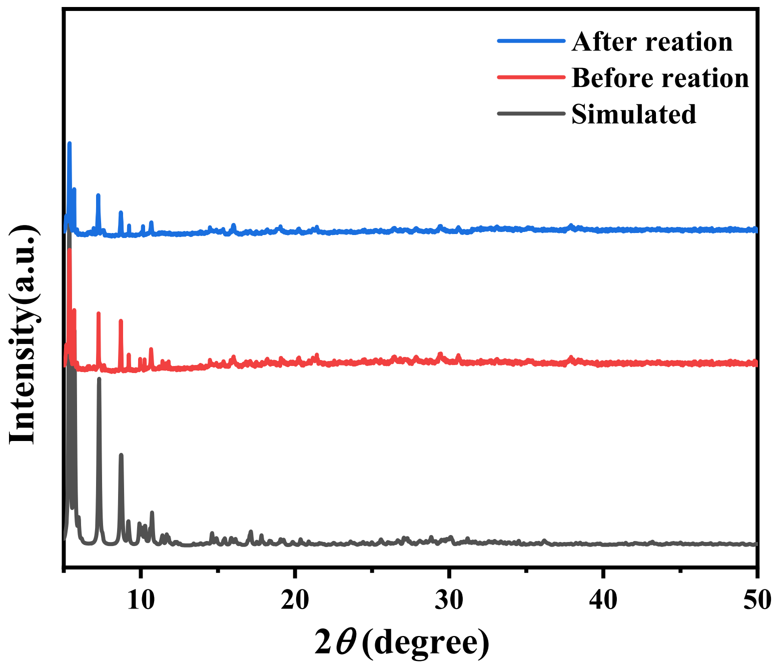

Fig. S9 PXRD patterns of **[Co@{Co$_{16}$Mo$_{16}$}]** before and after four photocatalytic reaction cycles.

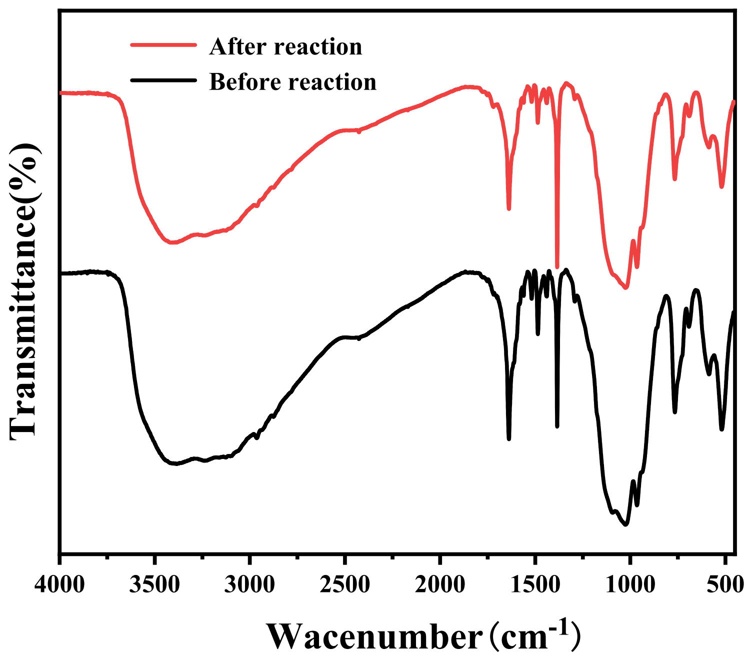

Fig. S10 IR spectra of **[Co@{Co$_{16}$Mo$_{16}$}]** before and after four photocatalytic reaction cycles.

4. CO_2 photocatalytic reduction

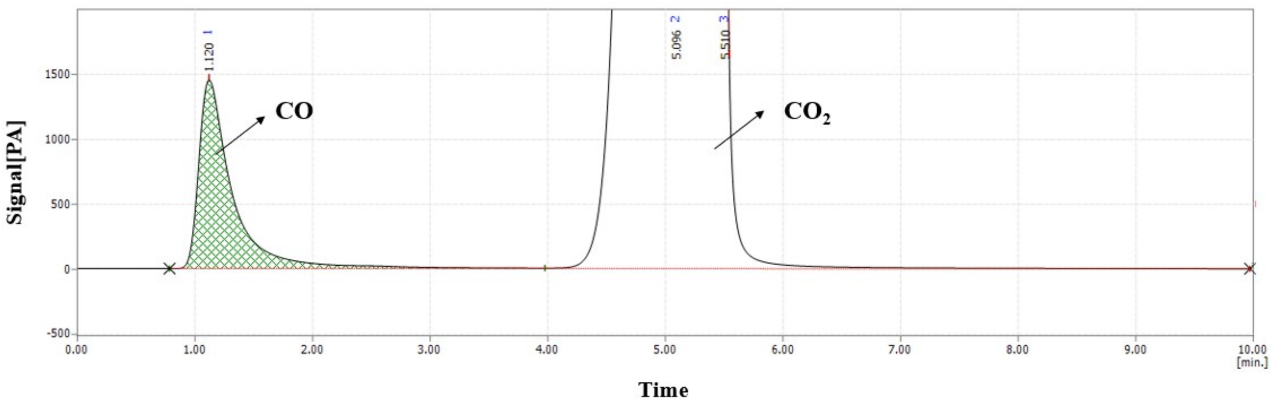

Fig. S11 GC profiles of CO_2 reduction to CO with **[Co@{$Co_{16}Mo_{16}$}]** as catalyst after reaction 8h.

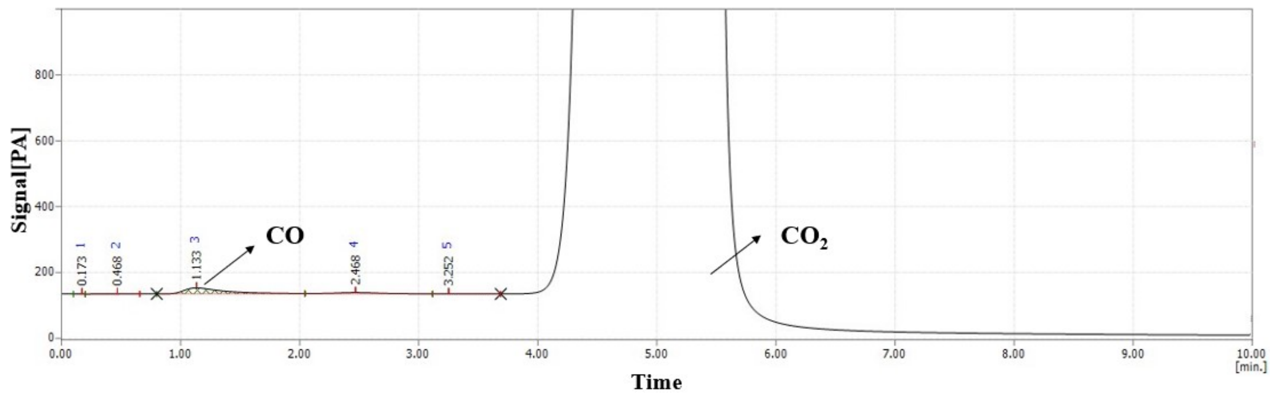

Fig. S12 GC profiles of CO_2 reduction to CO without catalyst after reaction 8h.

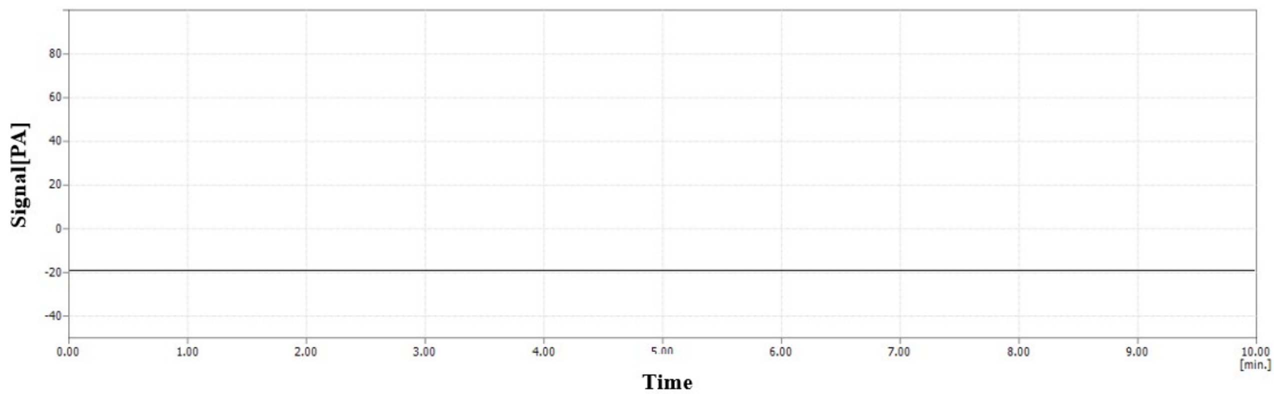

Fig. S13 The on-line curve of the produced H_2 detected by GC-TCD.

Table S3 Investigations on reaction conditions for **[Co@{$Co_{16}Mo_{16}$}]**.

Entry	CO (μmol)	CH_4 (μmol)	TON_{CO}	Formation $rate_{CO}$ (μmol/g/h)	Selectivity (%)
1[a]	179.57	5.76	227.168	6764.31	96.89
2[b]	none	none	–	–	–
3[c]	3.24	0.54	4.099	81.00	85.43
4[d]	0.065	0.494	0.082	1.63	11.63
5[e]	none	none	–	–	–
6[f]	148.50	11.56	7.40	3712.50	92.78
7[g]	2.21	0.64	0.062	69.06	77.50
8[h]	none	none	–	–	–
9[i]	none	none	–	–	–

[a]Reaction conditions: **[Co@{$Co_{16}Mo_{16}$}]** (5 mg, 0.79 μmol), $[Ru(bpy)_3]Cl_2{\cdot}6H_2O$ (11.3 mg), mixed solvent (50 mL, MeCN/TEOA, 4/1), reaction gas (CO_2), λ ≥ 420 nm, 6 °C, 8 h reaction time; TON_{CO} = n(CO)/n(catalyst); Formation rate = n(CO)/m(catalyst)/(reaction time); Selectivity = n(CO)/n($CO+CH_4$)×100%, where n(CO) and n(catalyst) were the amounts of CO (mol) and the catalyst (mol), respectively. [b]Dark condition. [c]No catalyst **[Co@{$Co_{16}Mo_{16}$}]**. [d]No $[Ru(bpy)_3]Cl_2{\cdot}6H_2O$. [e]No TEOA. [f]$Co(OAc)_2$ replaced **[Co@{$Co_{16}Mo_{16}$}]**. [g]MoO_3 replaced **[Co@{$Co_{16}Mo_{16}$}]**. [h]4-phenylpyridine replaced **[Co@{$Co_{16}Mo_{16}$}]**. [i]Ar replaced CO_2.

Table S4 Comparison of reported other POM-based photocatalysts for CO_2-to-CO conversion.

Photocatalyst	Photosensitizer Sacrificial agent	Main product	$Rate_{CO}$ (μmol g^{-1} h^{-1})	$Sectivity_{CO}$ (%)	TON	Ref.
$[Co@\{Co_{16}Mo_{16}\}]$	$[Ru(bpy)_3]Cl_2\cdot 6H_2O$ triethanolamine	CO	6764.3	96.89	90.3	**this work**
$[Co@\{Co_{16}Mo_{16}\}]$2D	$[Ru(bpy)_3]Cl_2\cdot 6H_2O$ triethanolamine	CO	1203.56	94.45	69.72	**this work**
$(C_2H_5OH)(C_3H_5N_2)_6[Co_3(H_6P_4Mo_6O_{31})_2]\cdot H_2O$	$[Ru(bpy)_3]Cl_2\cdot 6H_2O$ triethanolamine	CO	723.6	—	17.7	[4]
$(H_2bbi)_2\{[Co_2(bbi)][Co_{2.5}(H_2O)_4][H_9CoP_8Mo_{12}O_{62}]\}$ $4H_2O$	$[Ru(bpy)_3]Cl_2\cdot 6H_2O$ triethanolamine	CO	407.6	93.3	—	[5]
$Na_6[Co(H_2O)_2(H_2tib)]_2\{Co[Mo_6O_{15}(HPO_4)_4]_2\}\cdot 5H_2O$	— triethanolamine	CO	1.09	96.3	—	[6]
$Na_3[Co(H_2O)_3][Co_2(bib)]$-$(H_2bib)_{2.5}\{HCo[Mo_6O_{14}(OH)(HPO_4)_4]_2\}\cdot 4H_2O$	— triethanolamine	CO	0.94	96.4	—	[6]
$[Co_{2.67}(SiW_{12}O_{40})(H_2O)_4(Htrz)_4]\cdot Cl_{1.33}$	$[Ru(bpy)_3]Cl_2\cdot 6H_2O$ triethanolamine	CO	5235	52	55.31	[7]
$Na_{10}Co_4(H_2O)_2(PW_9O_{34})_2$@g-$C_3N_4$	Bipyridine + $CoCl_2$ triethanolamine	CO	107	94	—	[8]
$K_4Na_{28}[\{Co_4(OH)_3(VO_4)\}_4(SiW_9O_{34})_4]$ $66H_2O$	$[Ru(phen)_3](PF_6)_2$ triethanolamine	CO	0.084	—	10492	[9]
$[Co(en)_2]_6[V_{12}B_{18}O_{54}(OH)_6]\cdot 17H_2O$	$[Ru(bpy)_3]Cl_2\cdot 6H_2O$ triethanolamine	CO	5700	—	47.77	[10]
$H_{47}Na_2Co_4Mo_{24}(PO_4)_{11}O_{72}\cdot 15H_2O$	$[Ru(bpy)_3]Cl_2\cdot 6H_2O$ triethanolamine	CO	1848.3	75.4	75.3	[11]
$[Ni_6(trz)_2(Htrz)_{13}][H_4P_4Mo_{11}O_{50}]\cdot 7H_2O$	$[Ru(bpy)_3]Cl_2\cdot 6H_2O$ triethanolamine	CO	689.86	—	—	[12]
TCOF-MnMo6	no	CO	37.25	100	—	[13]

Table S5 ICP results of fresh catalyst, recovered catalyst and supernatant after reaction.

Sample	Co (mg/L)	Mo (mg/L)
Fresh catalyst	1.237	2.214
Recovered catalyst	1.199	2.107
Supernatant after reaction	-	0.067

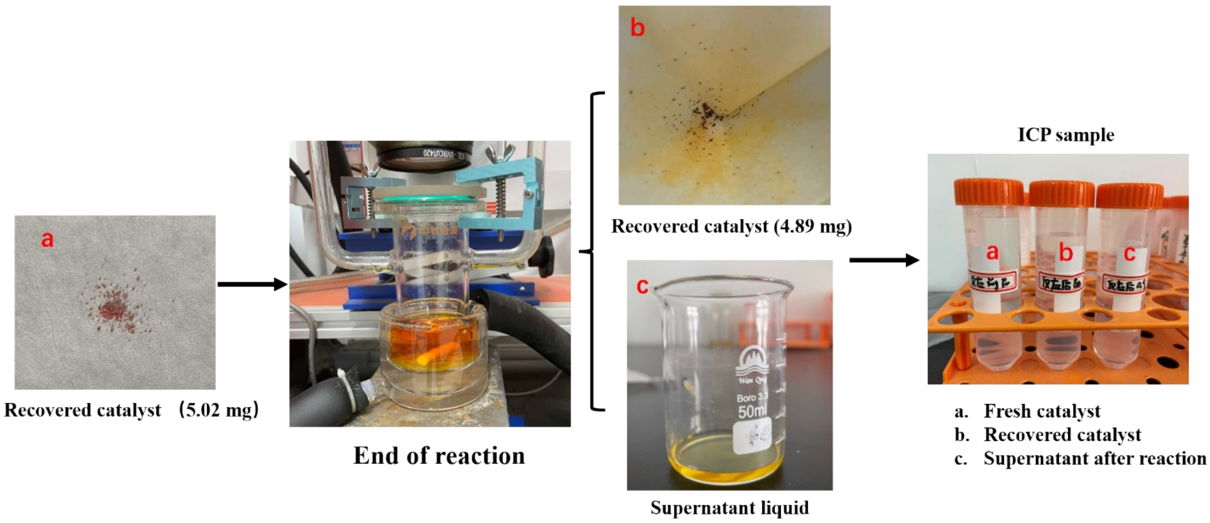

Fig. S14 Catalyst recovery and ICP sample preparation.

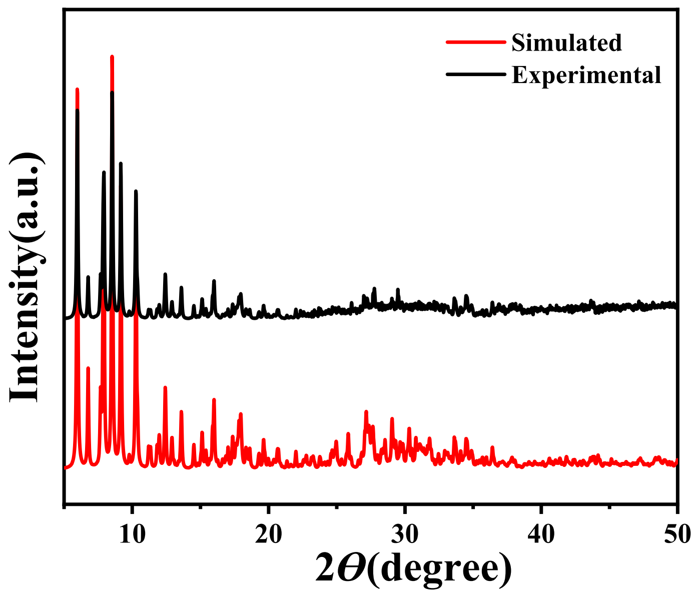

Fig. S15 PXRD patterns of **[Co@{$Co_{16}Mo_{16}$}]2D**.

5. Selected bond lengths and bond angles

Table S6 Selected bond lengths (Å) and bond angles (˚) for **[Co@{$Co_{16}Mo_{16}$}]**.

Mo(1)-O(3)	2.054(7)	O(3)-Mo(1)-O(9)	85.4(3)
Mo(1)-O(9)	2.099(6)	O(3)-Mo(1)-O(17)	79.7(3)
Mo(1)-O(10)	1.964(7)	O(9)-Mo(1)-O(17)	79.4(3)
Mo(1)-O(16)	1.932(6)	O(10)-Mo(1)-O(3)	88.9(3)
Mo(1)-O(17)	2.216(6)	O(10)-Mo(1)-O(9)	160.5(3)
Mo(1)-O(43)	1.674(7)	O(10)-Mo(1)-O(17)	81.2(2)
Mo(2)-O(1)	1.949(7)	O(16)-Mo(1)-O(3)	162.4(3)
Mo(2)-O(2)	1.943(6)	O(16)-Mo(1)-O(9)	86.6(3)
Mo(2)-O(7)	2.079(6)	O(16)-Mo(1)-O(10)	93.5(3)
Mo(2)-O(23)	2.116(6)	O(16)-Mo(1)-O(17)	83.4(3)
Mo(2)-O(31)	2.183(7)	O(43)-Mo(1)-O(3)	93.9(3)
Mo(2)-O(58)	1.675(8)	O(43)-Mo(1)-O(9)	95.9(3)
Mo(3)-O(5)	2.074(7)	O(43)-Mo(1)-O(10)	103.2(3)
Mo(3)-O(14)	2.171(7)	O(43)-Mo(1)-O(16)	102.4(3)
Mo(3)-O(18)	2.057(6)	O(43)-Mo(1)-O(17)	172.4(3)
Mo(3)-O(26)	1.959(6)	O(1)-Mo(2)-O(7)	88.8(3)
Mo(3)-O(28)	1.946(7)	O(1)-Mo(2)-O(23)	161.1(3)
Mo(3)-O(52)	1.687(7)	O(1)-Mo(2)-O(31)	81.4(3)
Mo(4)-O(12)	2.049(8)	O(2)-Mo(2)-O(1)	93.8(3)
Mo(4)-O(26)	1.956(7)	O(2)-Mo(2)-O(7)	162.4(3)
Mo(4)-O(28)	1.941(6)	O(2)-Mo(2)-O(23)	86.2(3)
Mo(4)-O(34)	2.421(7)	O(2)-Mo(2)-O(31)	83.4(3)
Mo(4)-O(57)	1.682(7)	O(7)-Mo(2)-O(23)	85.9(2)
Mo(4)-O(61)	2.056(6)	O(7)-Mo(2)-O(31)	79.8(3)
Mo(5)-O(10)	1.939(6)	O(23)-Mo(2)-O(31)	79.8(3)
Mo(5)-O(16)	1.941(6)	O(58)-Mo(2)-O(1)	103.6(3)
Mo(5)-O(19)	2.055(7)	O(58)-Mo(2)-O(2)	103.3(3)
Mo(5)-O(20)	2.456(6)	O(58)-Mo(2)-O(7)	92.9(3)
Mo(5)-O(29)	2.053(7)	O(58)-Mo(2)-O(23)	94.8(3)
Mo(5)-O(46)	1.678(7)	O(58)-Mo(2)-O(31)	171.2(3)
Mo(6)-O(6)	1.964(7)	O(5)-Mo(3)-O(14)	78.7(3)
Mo(6)-O(8)	2.086(6)	O(18)-Mo(3)-O(5)	84.4(3)
Mo(6)-O(21)	1.945(6)	O(18)-Mo(3)-O(14)	79.1(3)
Mo(6)-O(36)	2.070(6)	O(26)-Mo(3)-O(5)	89.5(3)
Mo(6)-O(42)	1.664(8)	O(26)-Mo(3)-O(14)	80.8(3)
Mo(6)-O(44)	2.182(7)	O(26)-Mo(3)-O(18)	159.8(3)

Mo(7)-O(1)	1.941(6)	O(28)-Mo(3)-O(5)	161.7(3)
Mo(7)-O(2)	1.933(6)	O(28)-Mo(3)-O(14)	84.3(3)
Mo(7)-O(34)	2.447(7)	O(28)-Mo(3)-O(18)	85.7(3)
Mo(7)-O(40)	2.043(6)	O(28)-Mo(3)-O(26)	94.5(3)
Mo(7)-O(50)	2.052(7)	O(52)-Mo(3)-O(5)	93.5(3)
Mo(7)-O(51)	1.659(7)	O(52)-Mo(3)-O(14)	171.5(3)
Mo(8)-O(6)	1.939(6)	O(52)-Mo(3)-O(18)	97.0(3)
Mo(8)-O(11)	2.057(7)	O(52)-Mo(3)-O(26)	102.6(3)
Mo(8)-O(20)	2.421(7)	O(52)-Mo(3)-O(28)	103.0(3)
Mo(8)-O(21)	1.926(6)	O(12)-Mo(4)-O(34)	83.1(3)
Mo(8)-O(56)	2.055(7)	O(12)-Mo(4)-O(61)	85.8(3)
Mo(8)-O(64)	1.663(8)	O(26)-Mo(4)-O(12)	163.3(3)
Co(1)-O(1W)	2.087(7)	O(26)-Mo(4)-O(34)	80.5(3)
Co(1)-O(10)	2.030(7)	O(26)-Mo(4)-O(61)	87.8(3)
Co(1)-O(24)	2.125(7)	O(28)-Mo(4)-O(12)	86.1(3)
Co(1)-O(41)	2.191(7)	O(28)-Mo(4)-O(26)	94.7(3)
Co(1)-O(48)	2.080(7)	O(28)-Mo(4)-O(34)	82.5(2)
Co(1)-O(54)	2.195(7)	O(28)-Mo(4)-O(61)	159.9(3)
Co(2)-O(1A)	2.232(7)	O(57)-Mo(4)-O(12)	94.4(3)
Co(2)-O(2W)	2.077(7)	O(57)-Mo(4)-O(26)	101.6(3)
Co(2)-O(15)	2.081(7)	O(57)-Mo(4)-O(28)	103.2(3)
Co(2)-O(16)	2.034(6)	O(57)-Mo(4)-O(34)	173.6(3)
Co(2)-O(59)	2.097(6)	O(57)-Mo(4)-O(61)	95.8(3)
Co(2)-O(62)	2.089(8)	O(61)-Mo(4)-O(34)	78.2(2)
Co(3)-O(3W)	2.083(8)	O(10)-Mo(5)-O(16)	94.0(3)
Co(3)-O(1)	2.052(7)	O(10)-Mo(5)-O(19)	88.1(3)
Co(3)-O(4)	2.187(7)	O(10)-Mo(5)-O(20)	79.4(2)
Co(3)-O(15)	2.097(7)	O(10)-Mo(5)-O(29)	162.5(3)
Co(3)-O(30)	2.182(7)	O(16)-Mo(5)-O(19)	159.3(3)
Co(3)-O(59)	2.108(7)	O(16)-Mo(5)-O(20)	82.7(3)
Co(4)-O(4W)	2.075(8)	O(16)-Mo(5)-O(29)	87.2(3)
Co(4)-O(6)	2.044(7)	O(19)-Mo(5)-O(20)	77.4(2)
Co(4)-O(22)	2.135(6)	O(29)-Mo(5)-O(19)	84.8(3)
Co(4)-O(41)	2.200(7)	O(29)-Mo(5)-O(20)	83.5(2)
Co(4)-O(47)	2.116(7)	O(46)-Mo(5)-O(10)	102.5(3)
Co(4)-O(54)	2.190(7)	O(46)-Mo(5)-O(16)	104.3(3)
Co(5)-O(5W)	2.091(8)	O(46)-Mo(5)-O(19)	95.3(3)
Co(5)-O(4)	2.191(7)	O(46)-Mo(5)-O(20)	172.5(3)
Co(5)-O(26)	2.021(6)	O(46)-Mo(5)-O(29)	94.1(3)
Co(5)-O(30)	2.267(8)	O(6)-Mo(6)-O(8)	89.5(3)

Co(5)-O(38)	2.147(7)	O(6)-Mo(6)-O(36)	161.4(3)
Co(5)-O(39)	2.087(7)	O(6)-Mo(6)-O(44)	80.1(3)
Co(6)-O(1A)	2.344(7)	O(8)-Mo(6)-O(44)	80.3(3)
Co(6)-O(6W)	2.081(9)	O(21)-Mo(6)-O(6)	93.3(3)
Co(6)-O(21)	2.045(6)	O(21)-Mo(6)-O(8)	163.3(3)
Co(6)-O(38)	2.037(7)	O(21)-Mo(6)-O(36)	85.7(3)
Co(6)-O(39)	2.073(7)	O(21)-Mo(6)-O(44)	84.0(3)
Co(6)-O(62)	2.048(7)	O(36)-Mo(6)-O(8)	86.4(3)
Co(7)-O(2A)	2.283(7)	O(36)-Mo(6)-O(44)	81.4(3)
Co(7)-O(7W)	2.101(7)	O(42)-Mo(6)-O(6)	102.7(3)
Co(7)-O(24)#1	2.072(6)	O(42)-Mo(6)-O(8)	92.9(3)
Co(7)-O(25)	2.088(7)	O(42)-Mo(6)-O(21)	102.5(3)
Co(7)-O(28)	2.034(7)	O(42)-Mo(6)-O(36)	95.6(3)
Co(7)-O(48)#1	2.077(7)	O(42)-Mo(6)-O(44)	172.6(3)
Co(8)-O(2A)	2.259(7)	O(1)-Mo(7)-O(34)	79.3(3)
Co(8)-O(8W)	2.094(9)	O(1)-Mo(7)-O(40)	162.2(3)
Co(8)-O(2)	2.060(6)	O(1)-Mo(7)-O(50)	87.7(3)
Co(8)-O(22)#1	2.072(6)	O(2)-Mo(7)-O(1)	94.4(3)
Co(8)-O(25)	2.074(7)	O(2)-Mo(7)-O(34)	82.4(3)
Co(8)-O(47)#1	2.100(7)	O(2)-Mo(7)-O(40)	87.2(3)
Co(9)-O(9W)	2.097(8)	O(2)-Mo(7)-O(50)	158.9(3)
Co(9)-O(9W)#1	2.097(8)	O(40)-Mo(7)-O(34)	83.3(3)
Co(9)-O(10W)#1	2.074(8)	O(40)-Mo(7)-O(50)	84.6(3)
Co(9)-O(10W)	2.074(8)	O(50)-Mo(7)-O(34)	77.4(3)
Co(9)-O(11W)#1	2.092(9)	O(51)-Mo(7)-O(1)	103.0(3)
Co(9)-O(11W)	2.092(9)	O(51)-Mo(7)-O(2)	104.2(3)
O(38)-Co(5)-O(4)	92.9(3)	O(51)-Mo(7)-O(34)	172.8(3)
O(38)-Co(5)-O(30)	159.3(3)	O(51)-Mo(7)-O(40)	93.8(3)
O(39)-Co(5)-O(5W)	84.8(3)	O(51)-Mo(7)-O(50)	95.7(3)
O(39)-Co(5)-O(4)	102.4(3)	O(6)-Mo(8)-O(11)	88.2(3)
O(39)-Co(5)-O(30)	90.3(3)	O(6)-Mo(8)-O(20)	80.2(3)
O(39)-Co(5)-O(38)	77.6(3)	O(6)-Mo(8)-O(56)	162.4(3)
O(6W)-Co(6)-O(1A)	174.2(3)	O(11)-Mo(8)-O(20)	78.2(2)
O(21)-Co(6)-O(1A)	98.3(3)	O(21)-Mo(8)-O(6)	94.7(3)
O(21)-Co(6)-O(6W)	86.3(3)	O(21)-Mo(8)-O(11)	159.7(3)
O(21)-Co(6)-O(39)	169.1(3)	O(21)-Mo(8)-O(20)	82.5(3)
O(21)-Co(6)-O(62)	97.0(3)	O(21)-Mo(8)-O(56)	86.1(3)
O(38)-Co(6)-O(1A)	84.4(3)	O(56)-Mo(8)-O(11)	85.2(3)
O(38)-Co(6)-O(6W)	98.4(3)	O(56)-Mo(8)-O(20)	82.6(3)
O(38)-Co(6)-O(21)	98.2(3)	O(64)-Mo(8)-O(6)	102.6(3)

O(38)-Co(6)-O(39)	80.5(3)	O(64)-Mo(8)-O(11)	95.5(3)
O(38)-Co(6)-O(62)	152.7(3)	O(64)-Mo(8)-O(20)	173.1(3)
O(39)-Co(6)-O(1A)	92.4(3)	O(64)-Mo(8)-O(21)	103.4(3)
O(39)-Co(6)-O(6W)	83.2(3)	O(64)-Mo(8)-O(56)	94.2(3)
O(62)-Co(6)-O(1A)	71.1(3)	O(1W)-Co(1)-O(24)	101.3(3)
O(62)-Co(6)-O(6W)	105.0(3)	O(1W)-Co(1)-O(41)	166.6(3)
O(62)-Co(6)-O(39)	88.6(3)	O(1W)-Co(1)-O(54)	94.5(3)
O(7W)-Co(7)-O(2A)	175.9(3)	O(10)-Co(1)-O(1W)	87.5(3)
O(24)#1-Co(7)-O(2A)	84.9(3)	O(10)-Co(1)-O(24)	92.9(3)
O(24)#1-Co(7)-O(7W)	99.2(3)	O(10)-Co(1)-O(41)	88.5(2)
O(24)#1-Co(7)-O(25)	154.8(3)	O(10)-Co(1)-O(48)	165.9(3)
O(24)#1-Co(7)-O(48)#1	79.4(3)	O(10)-Co(1)-O(54)	102.2(3)
O(25)-Co(7)-O(2A)	72.6(3)	O(24)-Co(1)-O(41)	91.7(3)
O(25)-Co(7)-O(7W)	103.3(3)	O(24)-Co(1)-O(54)	158.7(3)
O(28)-Co(7)-O(2A)	93.4(3)	O(41)-Co(1)-O(54)	73.9(3)
O(28)-Co(7)-O(7W)	86.4(3)	O(48)-Co(1)-O(1W)	83.7(3)
O(28)-Co(7)-O(24)#1	97.9(3)	O(48)-Co(1)-O(24)	78.1(3)
O(28)-Co(7)-O(25)	94.7(3)	O(48)-Co(1)-O(41)	102.5(3)
O(28)-Co(7)-O(48)#1	171.4(3)	O(48)-Co(1)-O(54)	89.5(3)
O(48)#1-Co(7)-O(2A)	94.5(3)	O(2W)-Co(2)-O(1A)	176.3(3)
O(48)#1-Co(7)-O(7W)	86.0(3)	O(2W)-Co(2)-O(15)	99.2(3)
O(48)#1-Co(7)-O(25)	91.0(3)	O(2W)-Co(2)-O(59)	85.6(3)
O(8W)-Co(8)-O(2A)	176.6(3)	O(2W)-Co(2)-O(62)	104.9(3)
O(8W)-Co(8)-O(47)#1	85.6(3)	O(15)-Co(2)-O(1A)	83.7(3)
O(2)-Co(8)-O(2A)	93.3(3)	O(15)-Co(2)-O(59)	79.8(3)
O(2)-Co(8)-O(8W)	86.0(3)	O(15)-Co(2)-O(62)	154.0(3)
O(2)-Co(8)-O(22)#1	97.3(3)	O(16)-Co(2)-O(1A)	90.4(3)
O(2)-Co(8)-O(25)	94.5(3)	O(16)-Co(2)-O(2W)	86.9(3)
O(2)-Co(8)-O(47)#1	171.0(3)	O(16)-Co(2)-O(15)	99.6(3)
O(22)#1-Co(8)-O(2A)	82.8(3)	O(16)-Co(2)-O(59)	172.3(3)
O(22)#1-Co(8)-O(8W)	100.5(3)	O(16)-Co(2)-O(62)	91.6(3)
O(22)#1-Co(8)-O(25)	153.9(3)	O(59)-Co(2)-O(1A)	97.2(3)
O(22)#1-Co(8)-O(47)#1	81.2(3)	O(62)-Co(2)-O(1A)	72.7(3)
O(25)-Co(8)-O(2A)	73.4(3)	O(62)-Co(2)-O(59)	92.1(3)
O(25)-Co(8)-O(8W)	103.4(3)	O(3W)-Co(3)-O(4)	167.8(3)
O(25)-Co(8)-O(47)#1	90.4(3)	O(3W)-Co(3)-O(15)	100.4(3)
O(47)#1-Co(8)-O(2A)	95.3(3)	O(3W)-Co(3)-O(30)	95.7(3)
O(9W)#1-Co(9)-O(9W)	180.0(4)	O(3W)-Co(3)-O(59)	85.1(3)
O(10W)#1-Co(9)-O(9W)	87.2(3)	O(1)-Co(3)-O(3W)	86.1(3)
O(10W)#1-Co(9)-O(9W)#1	92.8(3)	O(1)-Co(3)-O(4)	88.3(3)

O(22)-Co(4)-O(41)	90.8(3)	O(1)-Co(3)-O(15)	93.9(3)
O(22)-Co(4)-O(54)	158.3(3)	O(1)-Co(3)-O(30)	101.5(3)
O(47)-Co(4)-O(22)	79.4(2)	O(1)-Co(3)-O(59)	167.7(3)
O(47)-Co(4)-O(41)	103.0(3)	O(15)-Co(3)-O(4)	90.8(2)
O(47)-Co(4)-O(54)	89.1(3)	O(15)-Co(3)-O(30)	158.4(3)
O(54)-Co(4)-O(41)	73.9(2)	O(15)-Co(3)-O(59)	79.2(2)
O(5W)-Co(5)-O(4)	165.6(3)	O(30)-Co(3)-O(4)	74.8(2)
O(5W)-Co(5)-O(30)	94.7(3)	O(59)-Co(3)-O(4)	101.9(3)
O(5W)-Co(5)-O(38)	100.9(3)	O(59)-Co(3)-O(30)	87.9(3)
O(4)-Co(5)-O(30)	73.1(2)	O(4W)-Co(4)-O(22)	101.3(3)
O(26)-Co(5)-O(5W)	86.6(3)	O(4W)-Co(4)-O(41)	167.4(3)
O(26)-Co(5)-O(4)	88.7(3)	O(4W)-Co(4)-O(47)	82.8(3)
O(26)-Co(5)-O(30)	102.5(3)	O(4W)-Co(4)-O(54)	95.3(3)
O(26)-Co(5)-O(38)	92.1(3)	O(6)-Co(4)-O(4W)	87.6(3)
O(26)-Co(5)-O(39)	165.2(3)	O(6)-Co(4)-O(22)	93.1(3)
O(6)-Co(4)-O(47)	166.4(3)	O(6)-Co(4)-O(41)	88.3(3)
O(6)-Co(4)-O(54)	101.5(3)		

Symmetry transformations used to generate equivalent atoms: #1 -x, -y+1, -z+1

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