

## Electronic Supplementary Information

### **A S<sub>4</sub>N<sub>4</sub>-like [Co<sub>4</sub>(μ-Cl)<sub>4</sub>] based metal-organic framework with *sum* topology and selective CO<sub>2</sub> uptake**

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## 1. Materials and Measurements

All reagents were obtained from commercial sources and, unless otherwise noted, were used as received without further purification. Elemental analyses (C, H, and N) were performed on a Perkin-Elmer 240 analyzer.  $^1\text{H}$  NMR spectra were recorded on a Bruker DRX-500 spectrometer with tetramethylsilane as an internal reference. The IR spectra were obtained on a VECTOR TM 22 spectrometer with KBr pellets in the 4000- 400  $\text{cm}^{-1}$  region. Thermal gravimetric analyses (TGA) were performed under  $\text{N}_2$  atmosphere (100 mL/min) with a heating rate of 5  $^\circ\text{C}\cdot\text{min}^{-1}$  using a 2960 SDT thermogravimetric analyzer. Powder X-ray diffraction (PXRD) data were collected over the  $2\theta$  range 5  $\sim$  50 $^\circ$  on a Bruker D8 ADVANCE diffractometer using Cu  $K\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ) with a routine power of 1600 W (40 kV and 40 mA) in a scan speed of 0.2  $\text{s}\cdot\text{deg}^{-1}$  at room temperature. Using a Philip X' Pert Pro system, variable temperature PXRD (VT-PXRD) measurements were recorded after the sample had stayed at the respective temperature for 30 min in  $\text{N}_2$  atmosphere.

## 2. Experiment Details

**Synthesis of methyl 3,5-di(pyridin-4-yl)benzoate (HDPB).** Methyl 3,5-dibromobenzoate (2.0 g, 6.8 mmol), pyridin-4-ylboronic acid (2.5 g, 20.3 mmol), and  $\text{K}_3\text{PO}_4$  (15.0 g, 56.3 mmol) were added to 1,4-dioxane (120 mL). After stirring,  $\text{Pd}(\text{PPh}_3)_4$  (0.5 g, 0.4 mmol) was added, then the mixture was heated to 80  $^\circ\text{C}$  for 3 days under  $\text{N}_2$  atmosphere. The resultant was evaporated to dryness and taken up in  $\text{CH}_2\text{Cl}_2$  which later had been dried over  $\text{MgSO}_4$ . This  $\text{CH}_2\text{Cl}_2$  solution was evaporated to dryness and the residue was washed briefly with ethanol (20 mL). The crude product was hydrolyzed by refluxing in 2 M aqueous  $\text{NaOH}$  followed by acidification with 37 %  $\text{HCl}$  to afford HDPB. Yield = 1.5 g (78.9 %).  $^1\text{H}$  NMR (500 MHz,  $\text{DMSO}-d_6$ ,  $\delta$  ppm): 13.51 (s,  $\text{COOH}$ ), 8.79 (d, 4H,  $J = 5.0 \text{ Hz}$ , ArH), 8.50 (s, 1H, ArH), 8.43 (s, 2H, ArH), 8.07 (d, 4H,  $J = 5.0 \text{ Hz}$ , ArH). Anal. Calcd (Found) for  $\text{C}_{17}\text{H}_{12}\text{N}_2\text{O}_2$ : C, 73.90 (73.85); H, 4.38 (4.18); N, 10.14 (10.34) %. Selected IR (KBr,  $\text{cm}^{-1}$ ): 3381, 3064, 1715, 1633, 1609, 1595, 1553, 1507, 1384, 1217, 1068, 817, 777, 619.

**Synthesis of NJU-Bai18,  $[\text{Co}_4(\mu\text{-Cl})_4(\text{DPB})_4]\cdot 6\text{DMF}\cdot 8\text{H}_2\text{O}$ .** NJU-Bai 18 was synthesized by reaction of HDPB (10.0 mg, 0.04 mmol),  $\text{CoCl}_2\cdot 6\text{H}_2\text{O}$  (20 mg, 0.08 mmol) and 4 drops of concentrated nitric acid in 2 mL  $N,N'$ -dimethylformamide (DMF) and 0.5 mL MeOH at 70  $^\circ\text{C}$  for 3 days. The red block crystals were collected and washed with DMF to yield 10.0 mg (yield: 70 % based on the ligand). Anal. Calcd (Found) for  $\text{C}_{86}\text{H}_{102}\text{Cl}_4\text{Co}_4\text{N}_4\text{O}_{22}$ : C, 50.11 (50.52); H, 4.99 (5.13); N, 9.51 (9.39) %. Selected IR (KBr,  $\text{cm}^{-1}$ ): 3196, 1668, 1620, 1590, 1550, 1500, 1442, 1399, 1318, 1254, 1220, 1094, 1069, 1016, 832, 783, 657, 630

### 3. X-ray single crystal structure determination

X-ray diffraction single-crystal data were measured on a Bruker Apex II CCD diffractometer at 296 K using graphite monochromated Mo K $\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ). Data reduction was made with the Bruker SAINT program. The structure was solved by direct methods and refined with full-matrix least squares technique using the SHELXTL package<sup>1</sup>. Displacement parameters were refined anisotropically, and the positions of the hydrogen atoms were generated geometrically, assigned isotropic thermal parameters, and allowed to ride on their parent carbon atoms before the final cycle of refinement. Because guest solvent molecules were highly disordered and impossible to refine using conventional discrete-atom models, the SQUEEZE subroutine of the PLATON software suite<sup>2</sup> was applied to remove them from the structure.

A summary of the crystallographic data are given in Table S1. CCDC 1496172 contains the supplementary crystallographic data for NJU-Bai18. The data can be obtained free of charge at [www.ccdc.cam.ac.uk/conts/retrieving.html](http://www.ccdc.cam.ac.uk/conts/retrieving.html) or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK.

### 4. Low-Pressure gas sorption measurements

The solvent-exchanged sample was prepared by immersing the as-synthesized samples in dry MeOH for 3 days to remove the nonvolatile solvate, and the extract was decanted every 8 hours and fresh MeOH was replaced. The completely activated sample was obtained by heating the solvent-exchanged sample at 60 °C under a dynamic high vacuum for 20 hours.

Low-pressure gases (H<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub> and CO<sub>2</sub>) sorption isotherms (up to 1 bar) were performed on a Quantachrome Autosorb-iQ volumetric gas adsorption analyzer. Ultra-high-purity grade H<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>, CO<sub>2</sub> and He (99.999% purity) gases were used throughout the gas adsorption experiments.

### 5. Estimation of the isosteric heats of gas adsorption through the virial method

A virial-type<sup>3</sup> expression comprising the temperature-independent parameters  $a_i$  and  $b_j$  was employed to calculate the isosteric heats of adsorption for H<sub>2</sub> (at 77 and 87 K), CO<sub>2</sub> and CH<sub>4</sub> (at 273 and 298 K) on NJU-Bai18. In each case, the data were fitted using the equation:

$$\ln P = \ln N + 1/T \sum_{i=0}^m a_i N^i + \sum_{j=0}^n b_j N^j \quad (1)$$

Here,  $P$  is the pressure expressed in Torr,  $N$  is the amount adsorbed in mmol g<sup>-1</sup>,  $T$  is the temperature in K,  $a_i$  and  $b_j$  are virial coefficients, and  $m$ ,  $n$  represent the number of coefficients required to adequately describe the isotherms ( $m$  and  $n$  were gradually increased until the contribution of extra

added  $a$  and  $b$  coefficients was deemed to be statistically insignificant towards the overall fit and the average value of the squared deviations from the experimental values was minimized). The values of the virial coefficients  $a_0$  through  $a_m$  were then used to calculate the isosteric heats of adsorption using the following expression.

$$Q_{st} = -R \sum_{i=0}^m a_i N^i \quad (2)$$

$Q_{st}$  is the coverage-dependent isosteric heat of adsorption and  $R$  is the universal gas constant. The heat of CO<sub>2</sub>, CH<sub>4</sub> and H<sub>2</sub> sorption for NJU-Bai18 in this manuscript is determined by using the low pressure gas sorption data measured in the pressure range from 0 ~ 1 bar.

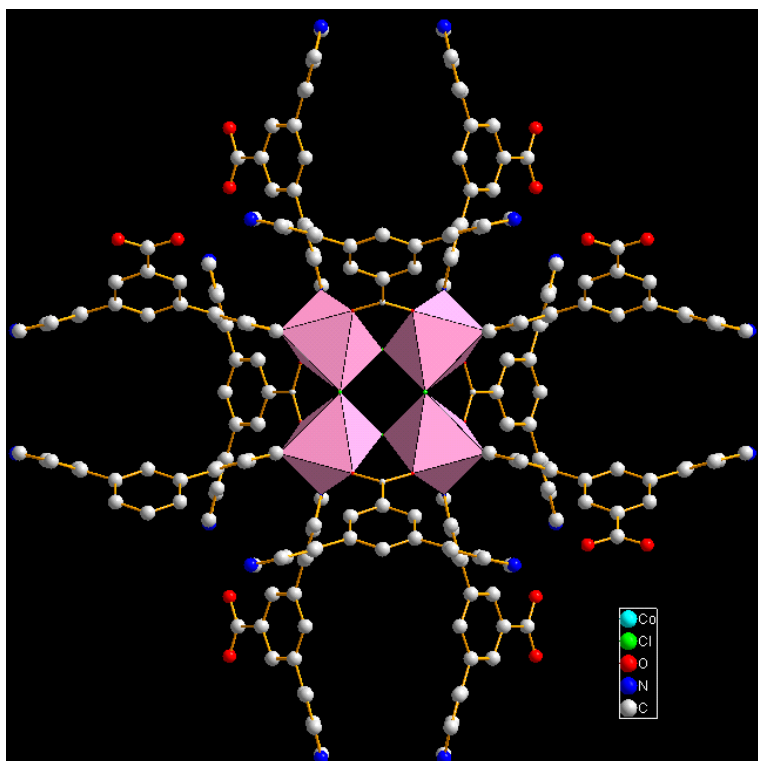
## 6. Binary mixture adsorption

IAST (ideal adsorption solution theory)<sup>4</sup> was used to predict binary mixture adsorption from the experimental pure-gas isotherms. In order to perform the integrations required by IAST, the single-component isotherms should be fitted by a proper model. The dual-site Langmuir-Freundlich equation was successful in fitting the data. As can be seen in Figure S15-16, the model fits the isotherms very well ( $R_2 > 0.9999$ ).

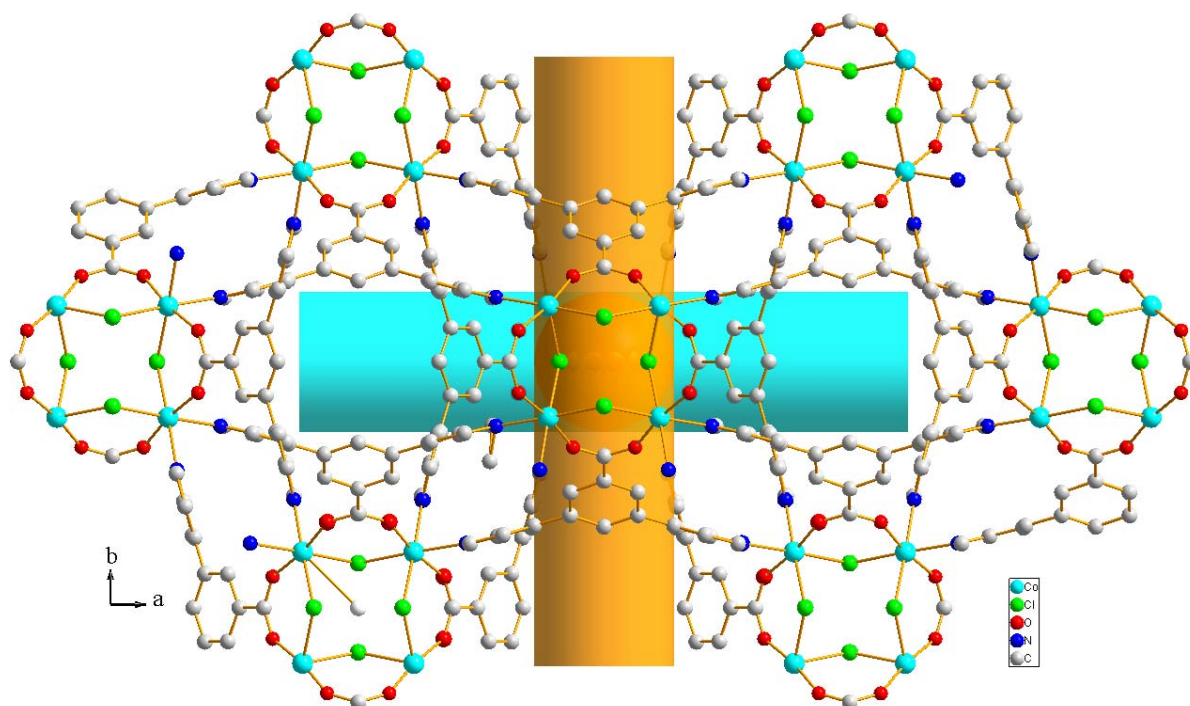
$$q = q_{m1} \cdot \frac{b_1 \cdot P^{1/n_1}}{1 + b_1 \cdot P^{1/n_1}} + q_{m2} \cdot \frac{b_2 \cdot P^{1/n_2}}{1 + b_2 \cdot P^{1/n_2}} \quad (3)$$

Here,  $P$  is the pressure of the bulk gas at equilibrium with the adsorbed phase (kPa),  $q$  is the adsorbed amount per mass of adsorbent (mol/kg),  $q_{m1}$  and  $q_{m2}$  are the saturation capacities of sites 1 and 2 (mol/kg),  $b_1$  and  $b_2$  are the affinity coefficients of sites 1 and 2 (1/kPa), and  $n_1$  and  $n_2$  represent the deviations from an ideal homogeneous surface.

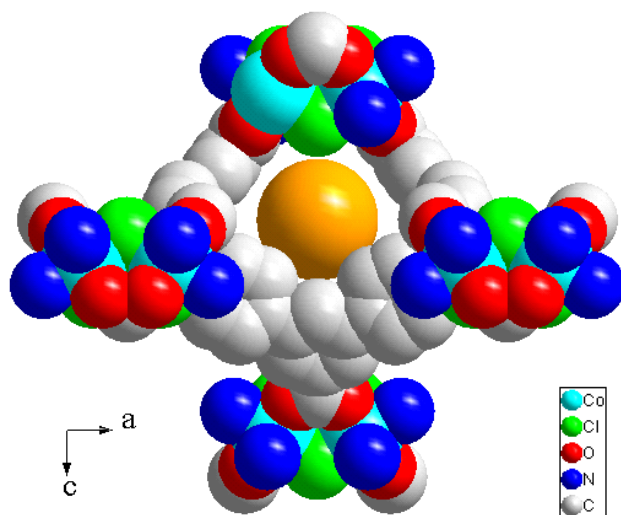
The selectivity  $S_{A/B}$  in a binary mixture of components A and B is defined as  $(x_A/y_A)/(x_B/y_B)$ , where  $x_i$  and  $y_i$  are the mole fractions of component  $i$  ( $i = A, B$ ) in the adsorbed and bulk phases, respectively.



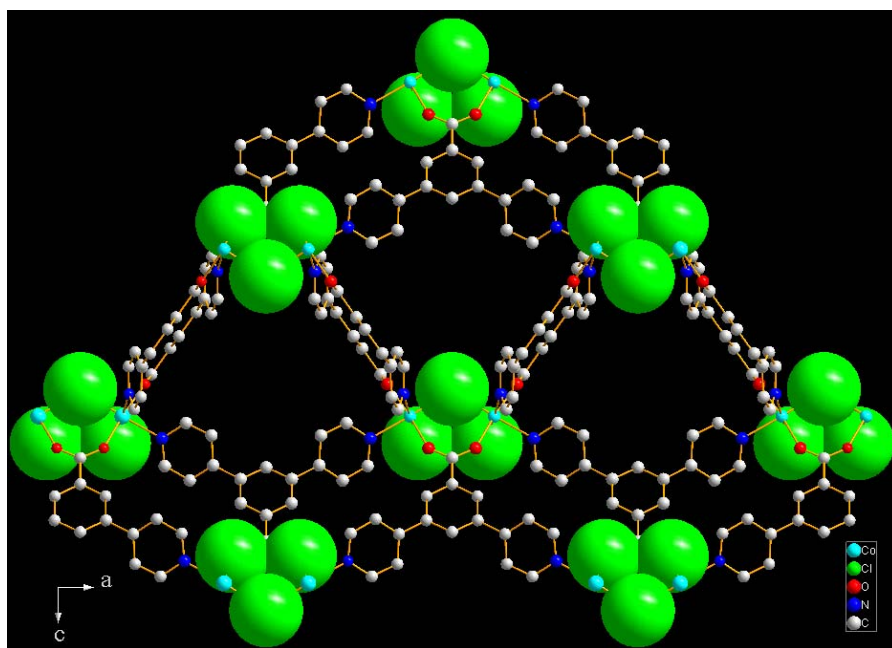
**Figure S1.** Perspective of the tetranuclear cobalt cluster in NJU-Bai18 coordinated by twelve ligands.



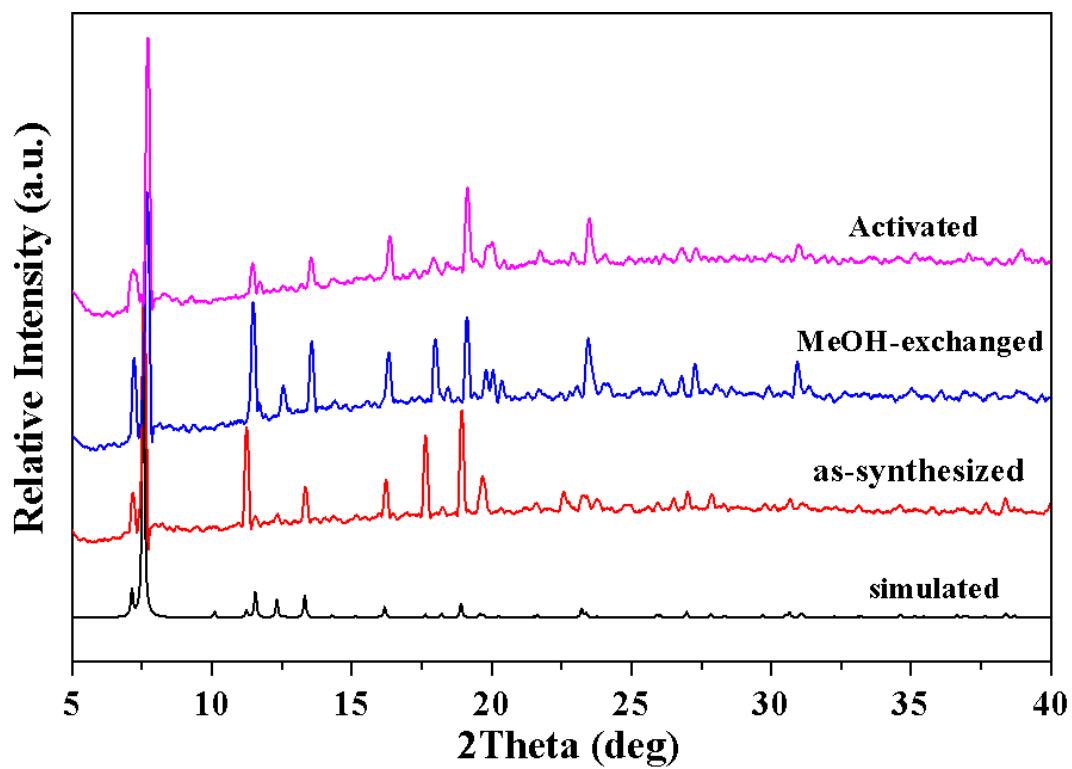
**Figure S2.** 3D network of NJU-Bai18 with two vertical channels formed along  $a/b$  axis (turquoise and orange cylinders, respectively).



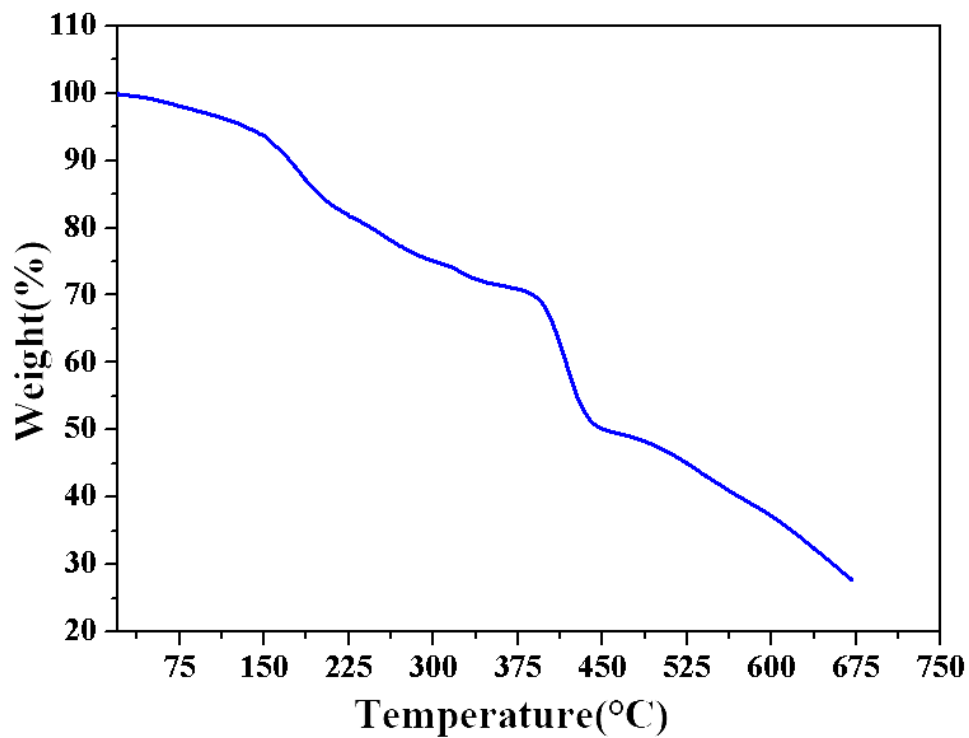
**Figure S3.** The open channel viewed along the  $b$  axis in NJU-Bai18.



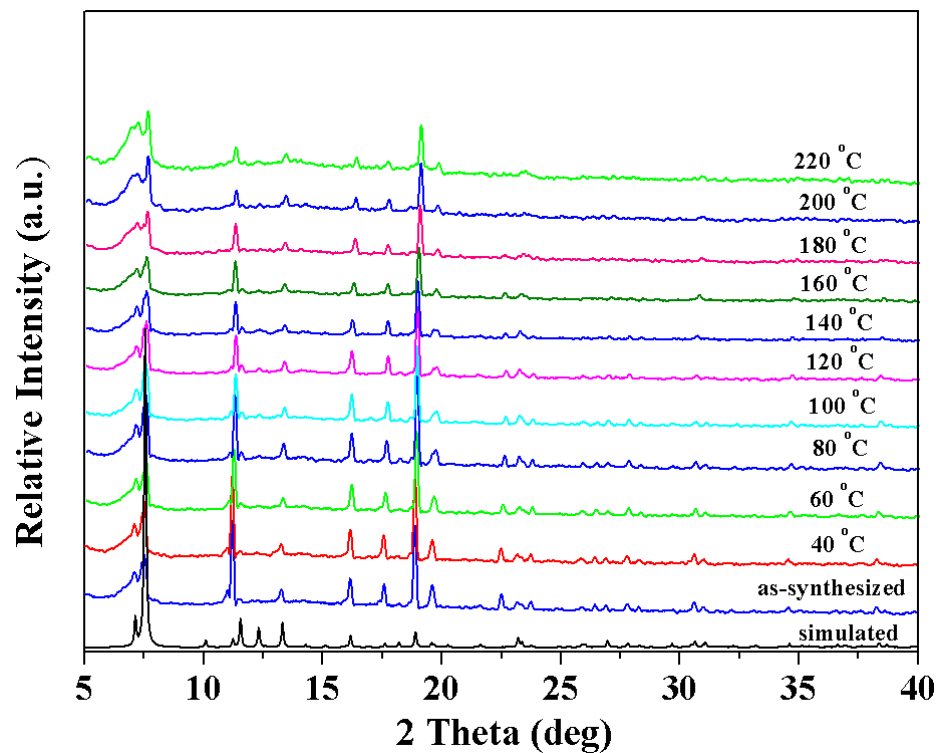
**Figure S4.** Channel along the  $b$  axis in NJU-Bai18 with the bridging chloride groups (space-filling mode) pointing into the channels.



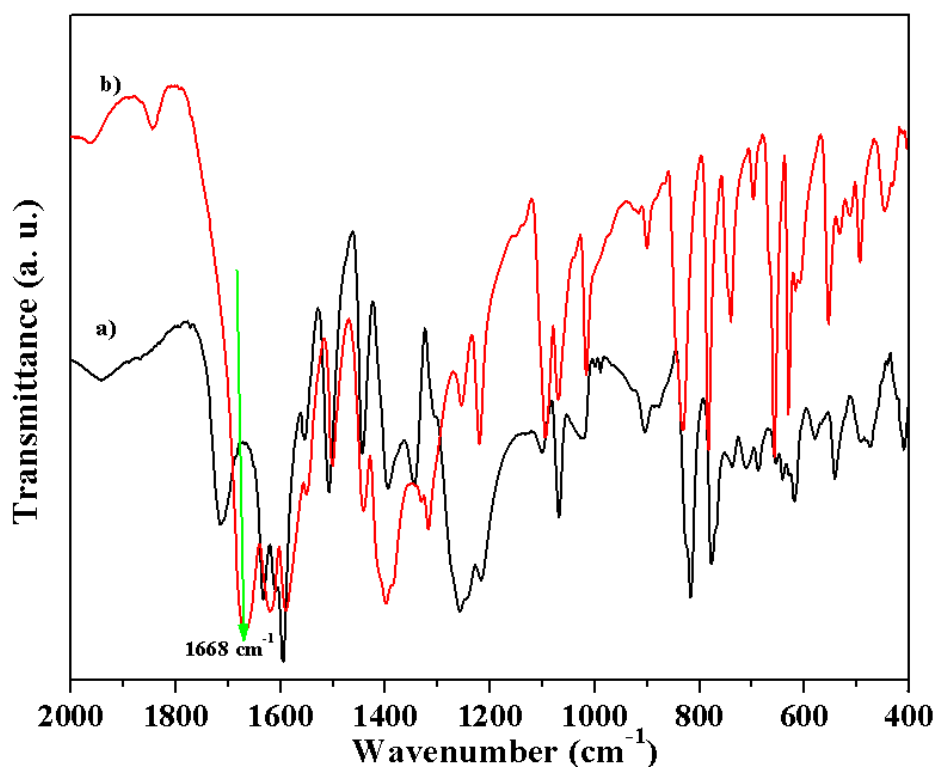
*Figure S5.* The PXRD patterns of NJU-Bai18.



*Figure S6.* TG curve of the as-synthesized sample of NJU-Bai18.

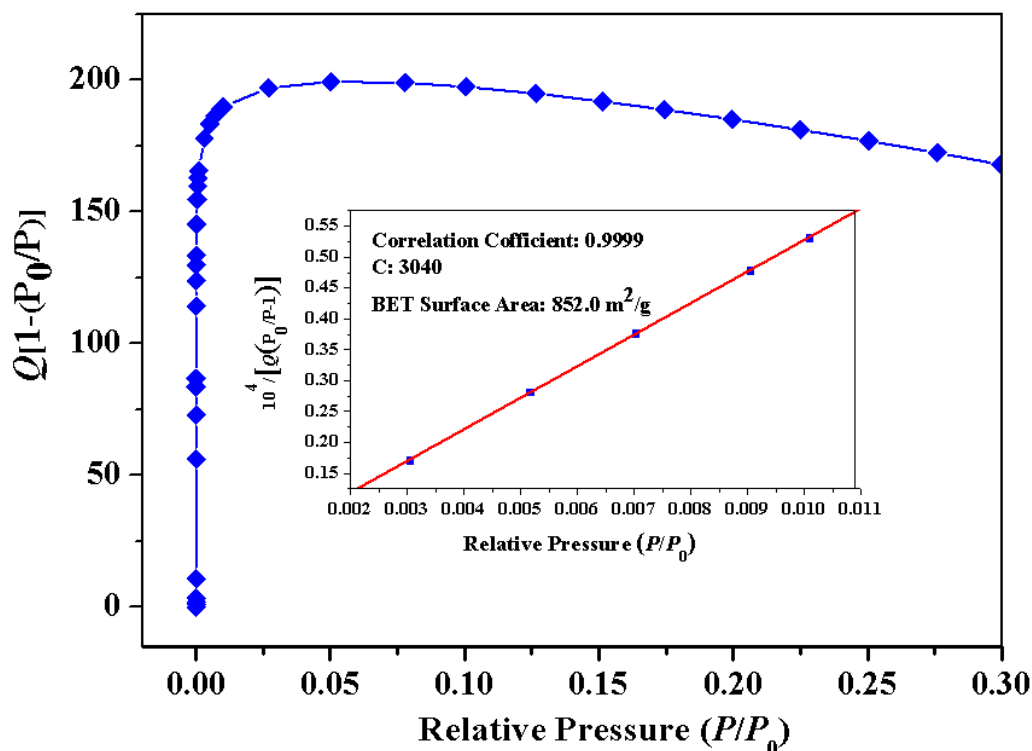


*Figure S7.* The VT-PXRD patterns of NJU-Bai18.

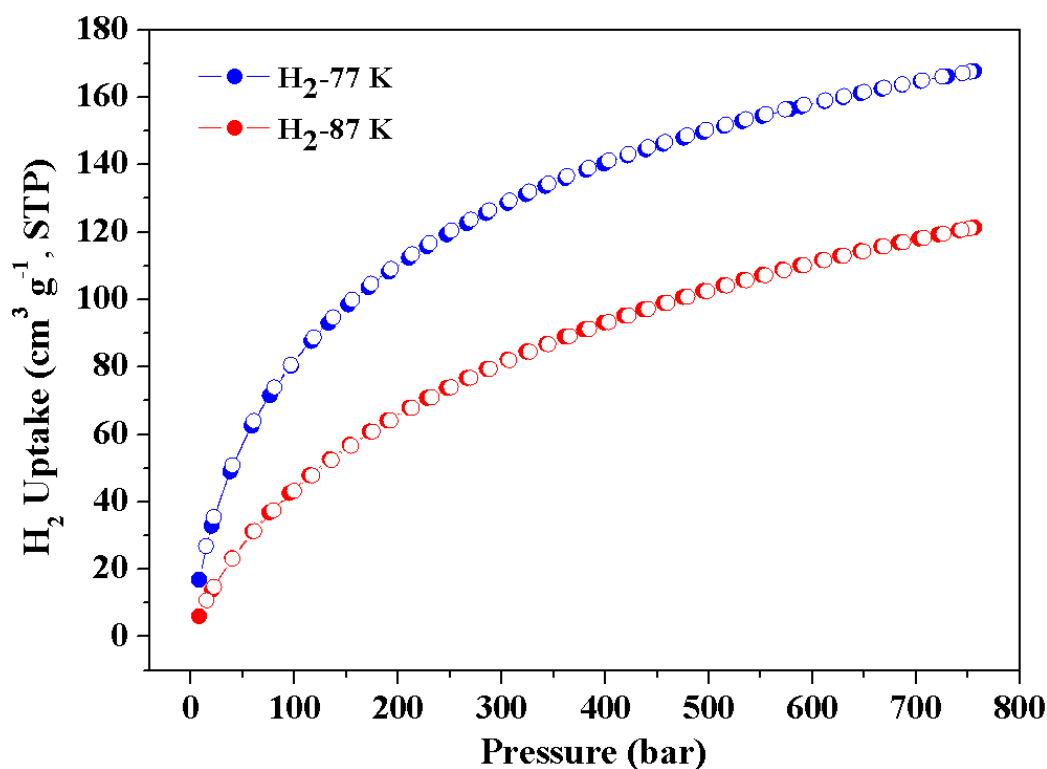


*Figure S8.* The IR spectra of NJU-Bai 18. (a) ligand of HDPB and (b) as-synthesized sample.





**Figure S9.** The BET plots for NJU-Bai18 in the chosen range ( $P/P_0 = 0.001-0.03$ ). This range was chosen according to two major criteria established in literatures<sup>5</sup>: (1) The pressure range selected should have values of  $Q(P_0-P)$  increasing with  $P/P_0$ . (2) The y intercept of the linear region must be positive to yield a meaningful value of the c parameter, which should be greater than zero.



**Figure S10.** The  $\text{H}_2$  adsorption isotherms of NJU-Bai18 in low pressure range (filled symbols, adsorption; open symbols, desorption).

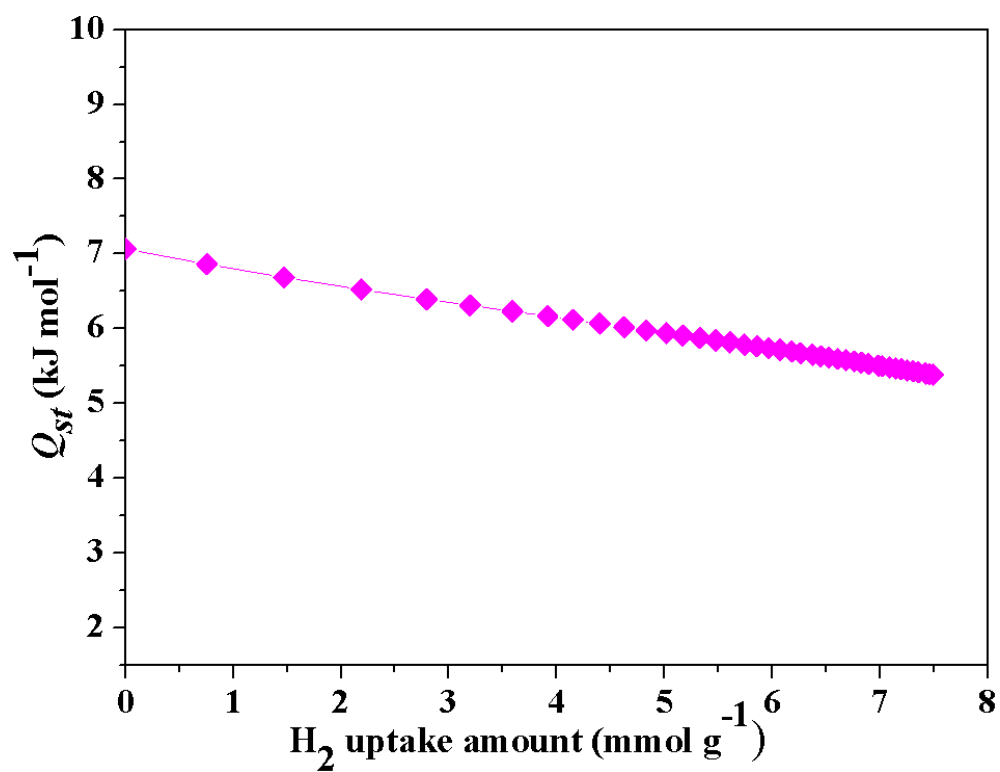


Figure S11. The H<sub>2</sub> adsorption enthalpy of NJU-Bai18.

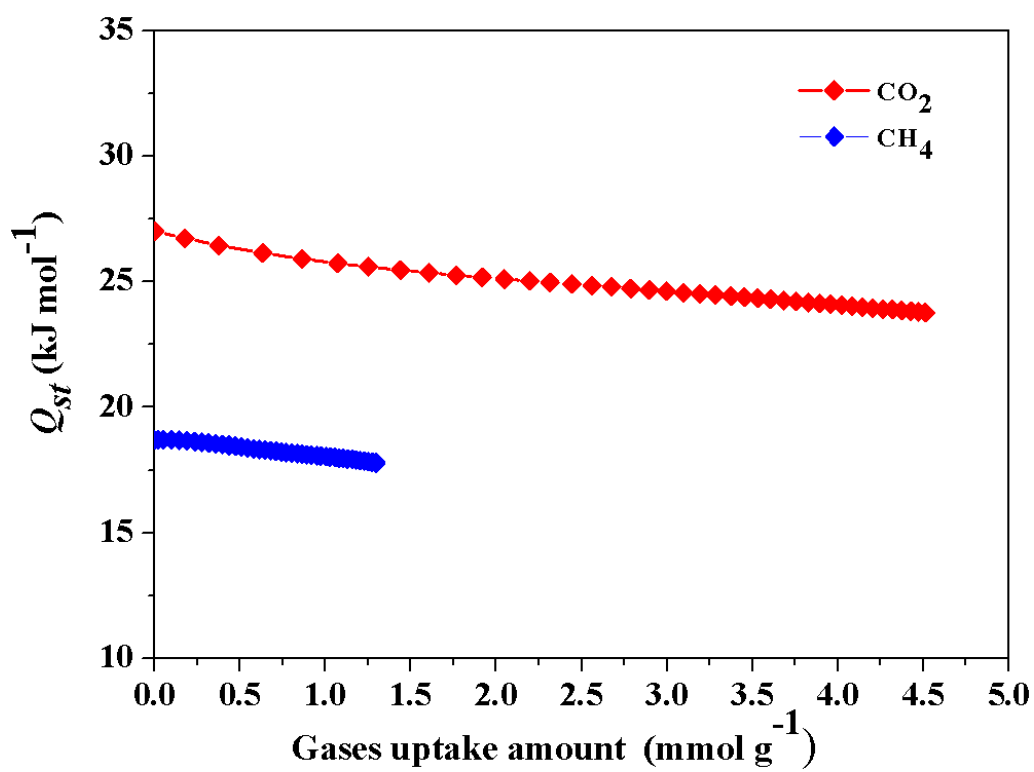
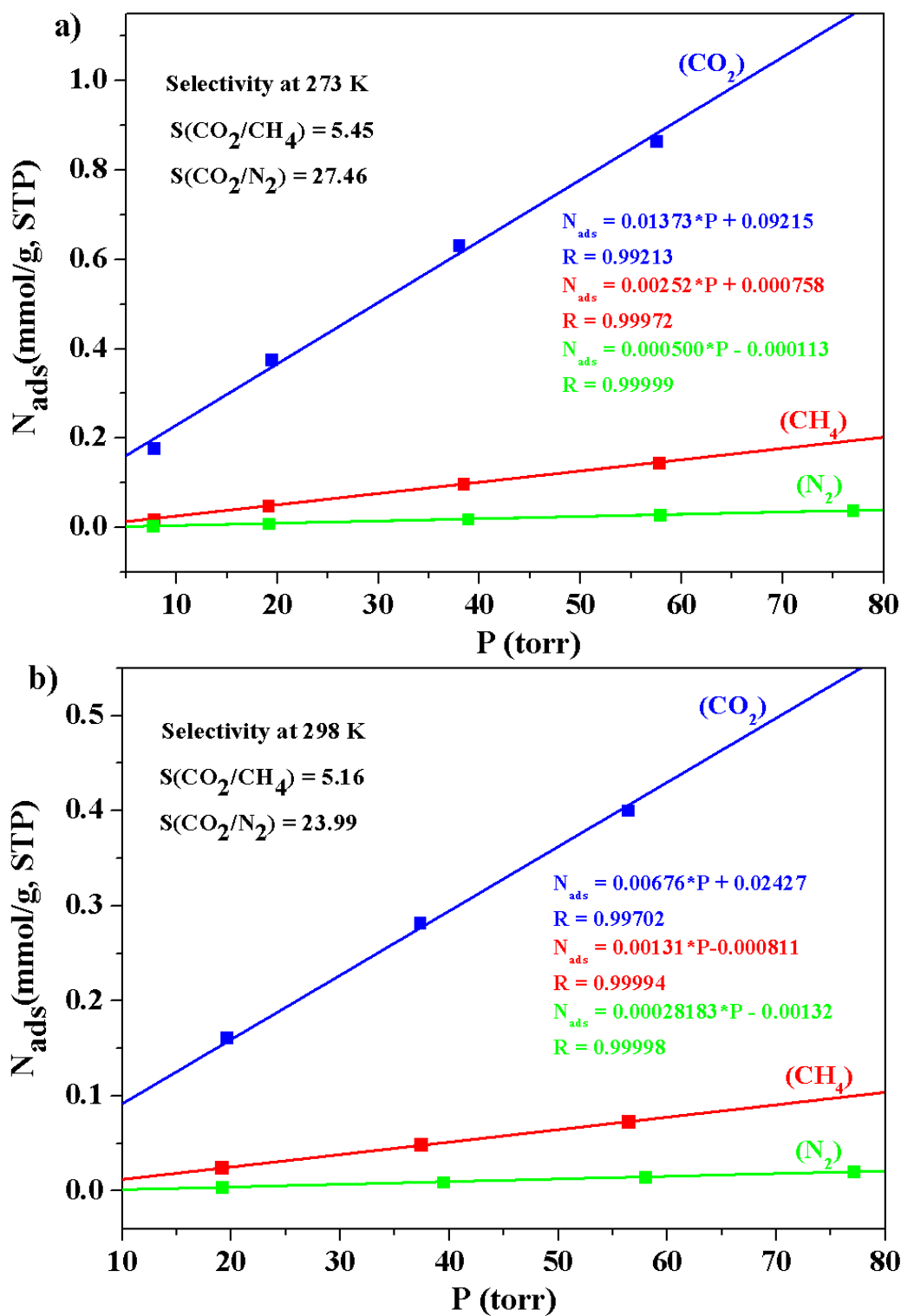
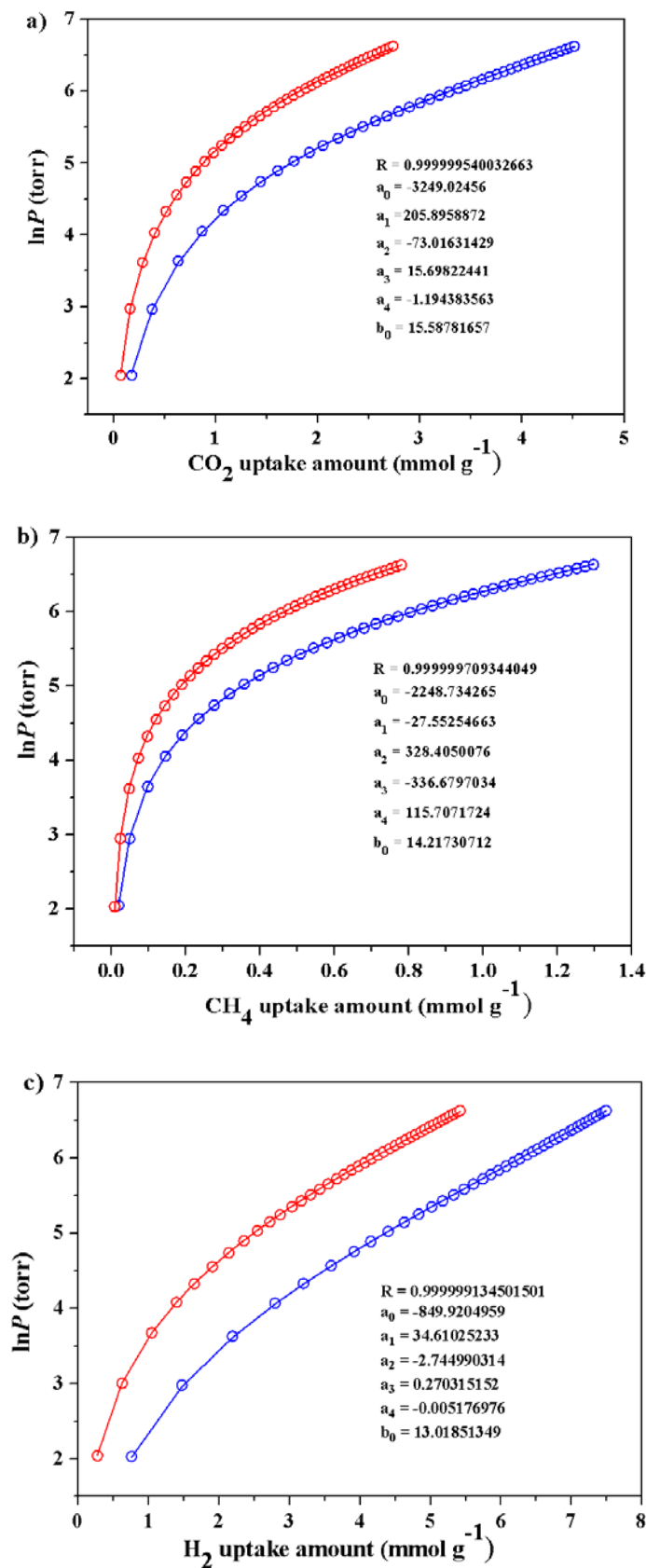


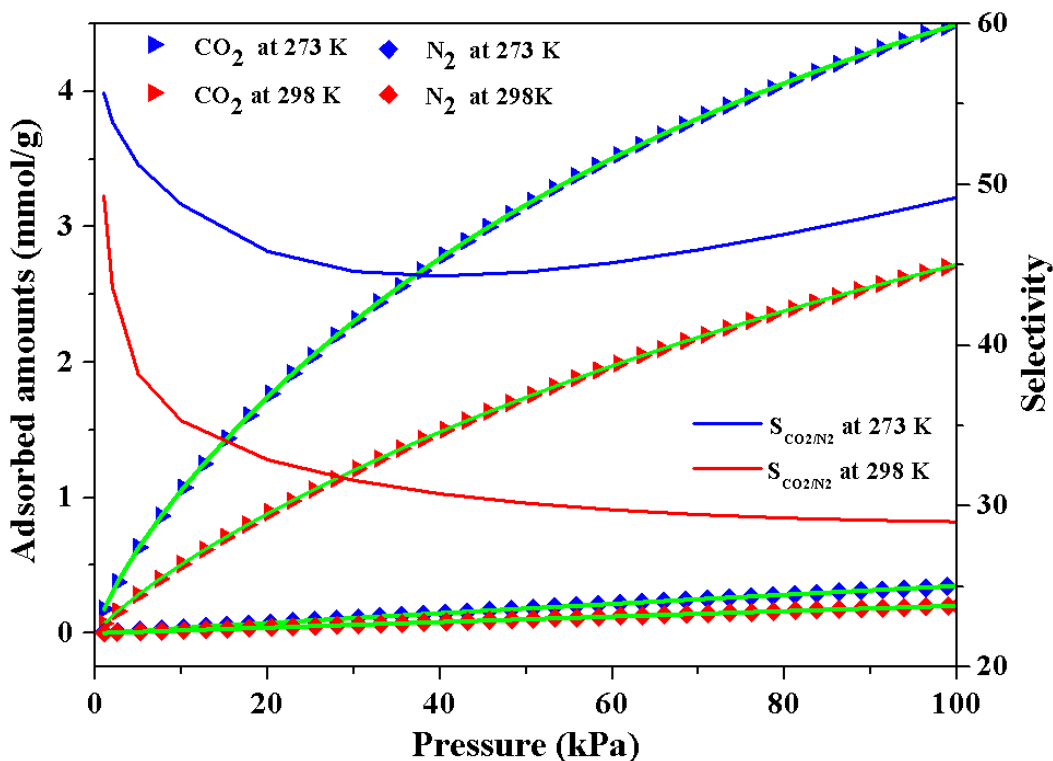
Figure S12. The CO<sub>2</sub> and CH<sub>4</sub> adsorption enthalpies of NJU-Bai18.



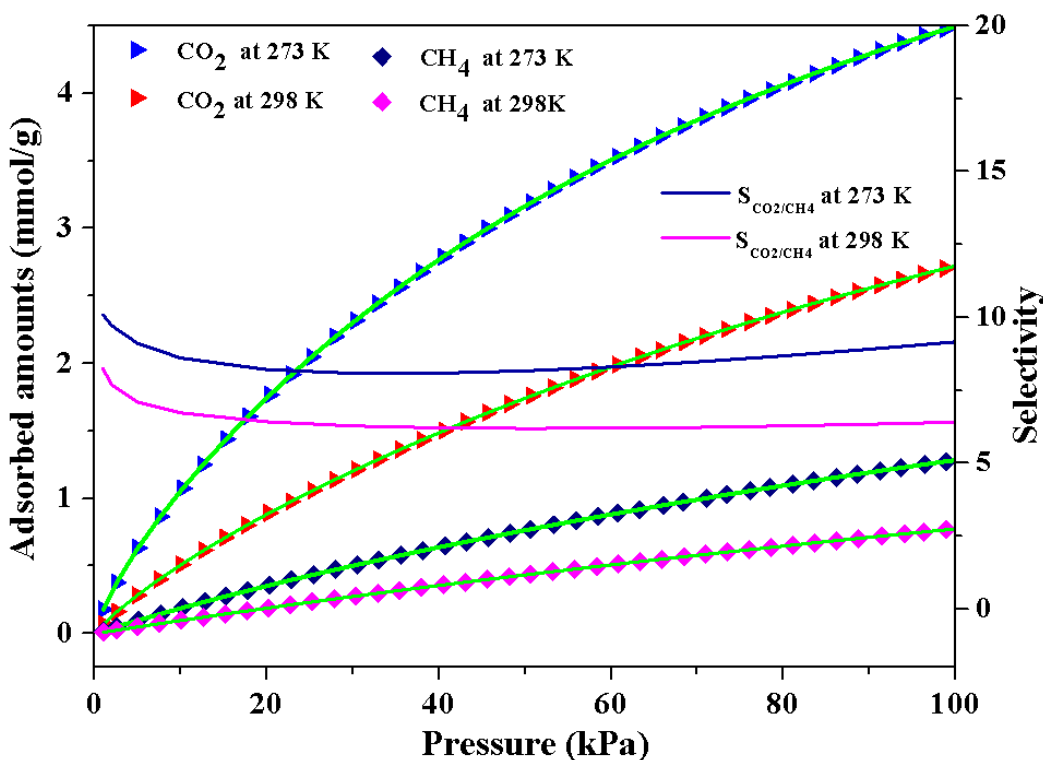
**Figure S13.** The fitting initial slope of CO<sub>2</sub>, CH<sub>4</sub> and N<sub>2</sub> isotherms for NJU-Bai18 collected at 273 K (a) and 298 K (b). ( $N_{\text{ads}}$  = gases uptake; R = related coefficient). The calculated selectivity of CO<sub>2</sub>/CH<sub>4</sub> and CO<sub>2</sub>/N<sub>2</sub> is 5.45, 27.46 and 5.16, 23.99 at 273 K and 298 K, respectively.



**Figure S14.** The gases isotherms (blue and red symbols, respectively) and the corresponding virial equation fits (blue and red lines, respectively) for NJU-Bai18. (a-b):  $\text{CO}_2$  and  $\text{CH}_4$  at 273 K and 298 K; (c):  $\text{H}_2$  at 77 K and 87 K.



**Figure S15.** The gases adsorption isotherms and the dual-site Langmuir-Freundlich fit lines of CO<sub>2</sub> and N<sub>2</sub> in NJU-Bai18 at 273 K and 298 K. The blue and red lines show the IAST predicted CO<sub>2</sub>/N<sub>2</sub> selectivity for a 15/85 molar mixture at 273 K and 298 K, respectively.



**Figure S16.** The gases adsorption isotherms and the dual-site Langmuir-Freundlich fit lines of CO<sub>2</sub> and CH<sub>4</sub> in NJU-Bai18 at 273 K and 298 K. The royal blue and magenta lines show the IAST predicted CO<sub>2</sub>/CH<sub>4</sub> selectivity for a 50/50 molar mixture at 273 K and 298 K, respectively.

**Table S1.** Crystal data and structure refinement for NJU-Bai18.

Identification code	NJU-Bai 18
CCDC number	1496172
Empirical formula	C <sub>17</sub> H <sub>11</sub> ClCoN <sub>2</sub> O <sub>2</sub>
Formula weight	369.66
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system	Tetragonal
Space group	P4 <sub>2</sub> /nmc
Unit cell dimensions	a = 17.5317(12), b = 17.5317(12), c = 15.757(2); α = β = γ = 90°
Volume	4843.1(8) Å <sup>3</sup>
Z	8
Density (calculated)	1.014 g·cm <sup>-3</sup>
Absorption coefficient	0.825 mm <sup>-1</sup>
F(000)	1496
Crystal size	0.20 × 0.20 × 0.16
Limiting indices	-20 ≤ h ≤ 16, -20 ≤ k ≤ 20, -18 ≤ l ≤ 15
Reflections collected / unique	26282 / 2291 [R(int) = 0.0318]
Completeness	100.0 % (theta = 25.02°)
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.876 and 0.848
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	2291 / 0 / 147
Goodness-of-fit on F <sup>2</sup>	1.061
Final R indices [I > 2σ(I)]	R <sub>1</sub> <sup>a</sup> = 0.0472, wR <sub>2</sub> <sup>a</sup> = 0.1633
R indices (all data)	R <sub>1</sub> <sup>a</sup> = 0.0526, wR <sub>2</sub> <sup>a</sup> = 0.1679
Largest diff. peak and hole	0.391 and -1.262 e·Å <sup>-3</sup>

$$^a R_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}; wR_2 = \left[ \frac{\sum w(F_o^2 - F_c^2)^2}{\sum w(F_o^2)^2} \right]^{1/2}$$

**Table S2.** Summary of the (3,12)-connected framework and corresponding Schläfli symbols.

Material	Schläfli symbols	Cluster-based SBU	Ref
[Co <sub>4</sub> (μ-Cl) <sub>4</sub> ]Cl <sub>4</sub> (DPB) <sub>4</sub> ·6DMF·8H <sub>2</sub> O ( <b>NJU-Bai18</b> )	(4 <sup>12</sup> ·6 <sup>34</sup> ·8 <sup>20</sup> )(4 <sup>3</sup> ) <sub>4</sub>	[Co <sub>4</sub> (μ-Cl) <sub>4</sub> ]	This work
[Cd(tipa)(μ <sub>3</sub> -OH)·NO <sub>3</sub> ·EtOH·DMF] <sub>n</sub>	(4 <sup>12</sup> ·6 <sup>42</sup> ·8 <sup>12</sup> )(4 <sup>3</sup> ) <sub>4</sub>	[Cd <sub>4</sub> (μ <sub>3</sub> -OH) <sub>4</sub> ]	6
[Co <sub>5</sub> (μ <sub>3</sub> -OH) <sub>2</sub> (5-NH <sub>2</sub> -bdc) <sub>4</sub> (H <sub>2</sub> O) <sub>2</sub> ]·7.5H <sub>2</sub> O	(4 <sup>19</sup> ·6 <sup>27</sup> ·8 <sup>20</sup> )(4 <sup>3</sup> ) <sub>4</sub>	[Co <sub>5</sub> (μ <sub>3</sub> -OH) <sub>4</sub> ]	7
[Zn <sub>9</sub> (BTB) <sub>4</sub> (odabco) <sub>3</sub> (μ <sub>3</sub> -O) <sub>3</sub> (μ-H <sub>2</sub> O) <sub>6</sub> ]·16DEF ( <b>FJI-2</b> )	(4 <sup>28</sup> ·6 <sup>34</sup> ·8 <sup>4</sup> ) <sub>3</sub> (4 <sup>3</sup> ) <sup>8</sup>	[Zn <sub>6</sub> (μ <sub>3</sub> -O) <sub>2</sub> (μ-H <sub>2</sub> O) <sub>4</sub> ]	8
[Na <sub>0.5</sub> Zn <sub>4.75</sub> (μ <sub>3</sub> -OH) <sub>2</sub> (BTB) <sub>2</sub> · (HBTB)(H <sub>2</sub> O) <sub>0.5</sub> ]·5DMF·1.5EtOH·10H <sub>2</sub> O ( <b>MCF-2</b> )	(4 <sup>3</sup> ) <sub>12</sub> (4 <sup>12</sup> ·6 <sup>36</sup> ·8 <sup>18</sup> ) <sub>2</sub> (4 <sup>12</sup> ·6 <sup>24</sup> ·8 <sup>30</sup> )	[NaZn <sub>6</sub> (μ <sub>3</sub> -OH) <sub>4</sub> (COO) <sub>12</sub> ]	9
[Zn <sub>8</sub> (μ <sub>3</sub> -OH) <sub>4</sub> (oba) <sub>6</sub> (bbi)(H <sub>2</sub> O) <sub>2</sub> ]	(3 <sup>2</sup> ·4) <sub>2</sub> (3 <sup>8</sup> ·4 <sup>22</sup> ·5 <sup>16</sup> ·6 <sup>18</sup> ·7 <sup>2</sup> )	[Zn <sub>8</sub> (μ <sub>3</sub> -OH) <sub>4</sub> ]	10
[Co <sub>8</sub> (μ <sub>3</sub> -OH) <sub>4</sub> (SO <sub>4</sub> ) <sub>2</sub> (dcpbpy) <sub>4</sub> (H <sub>2</sub> O) <sub>4</sub> ]· 12DMF·4EtOH·24H <sub>2</sub> O ( <b>MCF-32</b> )	(4 <sup>12</sup> ·6 <sup>34</sup> ·8 <sup>20</sup> )(4 <sup>3</sup> ) <sub>4</sub>	[Co <sub>8</sub> (μ <sub>3</sub> -OH) <sub>4</sub> (SO <sub>4</sub> ) <sub>2</sub> ]	11
[Ln(TTP) <sub>2</sub> ](CF <sub>3</sub> SO <sub>3</sub> ) <sub>3</sub> ·C <sub>3</sub> H <sub>6</sub> O·5H <sub>2</sub> O (Ln = Eu, Gd)	(4 <sup>20</sup> ·6 <sup>28</sup> ·8 <sup>18</sup> )(4 <sup>3</sup> ) <sub>4</sub>	Ln <sub>2</sub>	12
[Ln <sub>4</sub> (OH) <sub>4</sub> (3-SBA) <sub>4</sub> (H <sub>2</sub> O) <sub>4</sub> ]·nH <sub>2</sub> O (Ln = Eu, Gd, Tb)	(4 <sup>20</sup> ·6 <sup>28</sup> ·8 <sup>18</sup> )(4 <sup>3</sup> ) <sub>4</sub>	[Ln <sub>4</sub> (μ-OH) <sub>4</sub> ]	13
[Ln <sub>4</sub> (OH) <sub>4</sub> (SO <sub>4</sub> ) <sub>4</sub> (H <sub>2</sub> O) <sub>3</sub> ] (Ln = Y, Er)	(4 <sup>20</sup> ·6 <sup>28</sup> ·8 <sup>18</sup> )(4 <sup>3</sup> ) <sub>4</sub>	[Ln <sub>4</sub> (μ-OH) <sub>4</sub> (SO <sub>4</sub> ) <sub>4</sub> (H <sub>2</sub> O) <sub>3</sub> ]	14
{[Ln <sub>6</sub> (μ <sub>6</sub> -O)(μ <sub>3</sub> -OH) <sub>8</sub> (μ <sub>4</sub> -ClO <sub>4</sub> ) <sub>4</sub> (H <sub>2</sub> O) <sub>6</sub> ](OH) <sub>4</sub> ] <sub>n</sub> (Ln = Gd, Dy)	(4 <sup>20</sup> ·6 <sup>26</sup> ·8 <sup>20</sup> )(4 <sup>3</sup> ) <sub>4</sub>	[Ln <sub>6</sub> (μ <sub>6</sub> -O)(μ <sub>3</sub> -OH) <sub>8</sub> ]	15
[Eu <sub>6</sub> (μ <sub>6</sub> -O)(μ <sub>3</sub> -OH) <sub>8</sub> (H <sub>2</sub> O) <sub>6</sub> (SO <sub>4</sub> ) <sub>4</sub> ] <sub>n</sub>	(4 <sup>3</sup> ) <sub>4</sub> (4 <sup>20</sup> ·6 <sup>28</sup> ·8 <sup>18</sup> )	[Ln <sub>6</sub> (μ <sub>6</sub> -O)(μ <sub>3</sub> -OH) <sub>8</sub> ]	16

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