Electronic Supplementary Information

A window-space-directed assembly strategy for the construction of supertetrahedron-based zeolitic mesoporous metal-organic frameworks with ultramicroporous apertures for selective gas adsorption

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Materials and instrumentation

The solvents and reagents were purchased from commercial sources and used without purification. Thermogravimetric analysis (TGA) was performed with a TGA/DSC 1 STAR^e system at a heating rate of 5 °C/min under nitrogen atmosphere. Powder X-ray diffraction (PXRD) measurements were carried out at room temperature on a Bruker D8 Advance diffractometer at 40 kV, 40 mA with Cu *Ka* radiation ($\lambda = 0.154$ nm). In situ variable temperature PXRD patterns were collected on a Rigaku Ultima IV diffractometer. Scanning electron microscopy (SEM) images were collected using FEI Nova NanoSEM 450 scanning electron microscopes. The X-ray photoelectron spectroscopy (XPS) data were recorded on Thermo Scientific K-Alpha X-ray photoelectron spectrometer. The energy dispersive X-ray (EDX) spectroscopy was performed on samples loaded on a carbonfilm-coated Cu grid with the aid of an Oxford X-Max^N field-emission scanning electron microscope. Single component gas adsorption measurements were collected in the Accelerated Surface Area and Porosimetry 2020 System (ASAP2020).

Synthesis procedure

Synthesis of [Co₃(OH)(H₂O)(btc)₂(tpt)_{2/3}] (ST-sod-Co)

 $CoSO_4 \cdot 7H_2O$ (0.0633 g, 0.225 mmol), 2,4,6-tri(4-pyridyl)-1,3,5-triazine (tpt) (0.0155 g, 0.050 mmol) and 1,3,5-benzenetricarboxylate (H₂btc) (0.0315 g, 0.150 mmol) were added in the solvent mixture of N,N-Dimethylformamide (DMF, 4 mL)/methanol (CH₃OH, 1 mL) solvent in a 20 mL screw-capped vial, then 200µL HBF₄ (Tetrafluoroboric acid, 50 wt.% in water) was added into the solution and the mixture was sonicated for 30 min. Finally, the mixture was sealed and heated to 120 °C for 5 days. The red cubic crystals of ST-**sod**-Co were filtered and washed with DMF.

Synthesis of [Ni₃(OH)(H₂O)(btc)₂(tpt)_{2/3}] (ST-sod-Ni)

Ni(NO₃)₂·6H₂O (0.1308 g, 0.45 mmol), 2,4,6-tri(4-pyridyl)-1,3,5-triazine (tpt) (0.0310 g, 0.10 mmol) and 1,3,5-benzenetricarboxylate (H₂btc) (0.0632 g, 0.30 mmol) were added in the solvent mixture of N,N-Dimethylformamide (DMF, 3 mL)/ N,N-dimethylacetamide (DMA, 3 mL)/methanol (CH₃OH, 1 mL) solvent in a 20 mL screw-capped vial, then 400μ L H₂SiF₆ (Fluorosilicic acid , 25 wt.% in water) was added into the solution and the mixture was sonicated for 30 min. Finally, the mixture was sealed and heated to 120 °C for 5 days. The green cubic crystals of ST-**sod**-Ni were filtered and washed with

DMA.

Synthesis of [Co₂Ti(O)(H₂O)(btc)₂(tpt)_{2/3}] (ST-sod-Co/Ti)

 $CoCl_2 \cdot 6H_2O$ (0.0714 g, 0.30 mmol), Titanocene dichloride (Cp₂TiCl₂) (0.0375 g, 0.15 mmol), 2,4,6tri(4-pyridyl)-1,3,5-triazine (tpt) (0.0310 g, 0.10 mmol) and 1,3,5-benzenetricarboxylate (H₂btc) (0.0632 g, 0.30 mmol) were added in the solvent mixture of N,N-Dimethylformamide (DMF, 3 mL)/ N,N-dimethylacetamide (DMA, 3 mL)/methanol (CH₃OH, 1 mL) solvent in a 20 mL screw-capped vial, then 400µL H₂SiF₆ (Fluorosilicic acid , 25 wt.% in water) was added into the solution and the mixture was sonicated for 30 min. Finally, the mixture was sealed and heated to 120 °C for 5 days. The red polyhedral crystals of ST-**sod**-Co/Ti were filtered and washed with DMA.

For all above ST-**sod**-MOFs, the additive HBF_4 or H_2SiF_6 is necessary. The absence or replacement of the corresponding additive will lead to clear solution, unidentified powder, or impure samples. In addition, the change of metal sources also leads to amorphous or poorly crystalline materials under equivalent reaction conditions.

Crystallographic Study

Crystallographic data of ST-**sod**-Co was collected on Rigaku Oxford Diffraction Supernova diffractometer (graphite-monochromated Cu-K α radiation, λ = 1.54184 Å) at 100 K. Crystallographic data of ST-**sod**-Ni and ST-**sod**-Co/Ti were collected on Hybrid Pixel Array detector equipped with Ga-K α radiation (λ = 1.34050 Å) at 100 K. The structure was solved by direct methods and refined on F^2 by full-matrix, least-squares methods using the SHELXL-2014 program package. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms for the ligands were placed in calculated positions and treated as riding on their parents. The contribution of disordered solvent molecules was treated as diffuse using the SQUEEZE procedure implemented in PLATON, subsequently a set of solvent-free diffraction intensities was produced. It should be noted here that the tpt ligand is disordered over two positions (occupancy 0.5:0.5) in both structures ST-**sod**-Ni and ST-**sod**-Co/Ti. This is because the disorder of the triazine group leads to the carbon and nitrogen atoms sharing the same crystallographic positions with partial occupancies. The crystallographic refinement details for **ST-sod**-**MOFs** were given in table S1. Complete details can be found in the accompanying cif file. The following crystal structures have been deposited at the Cambridge Crystallographic Data Centre and

the CCDC number of 2026913, 2026914 and 2026915 are for ST-**sod**-Co, ST-**sod**-Ni and ST-**sod**-Co/Ti, respectively.

Gas adsorption experiments

Gas adsorption measurements were performed on the Accelerated Surface Area and Porosimetry 2020 System (ASAP2020) and pore-size analyzer. The solvent-exchange samples of ST-**sod**-MOFs were obtained by soaking the fresh crystalline samples in methanol for three days with methanol refreshing every 12 hours, and then the methanol was replaced by dichloromethane in the same process for another three days. Before each adsorption experiment, the solvent-exchange samples were degassed at 30 °C for 3 hours and further dried at 100 °C under high vacuum (<5 μ m Hg) for 15 hours to obtain the completely activated ST-**sod**-MOFs. The temperatures were controlled by a liquid nitrogen bath (77 K), an ice water bath (273 K) and a water bath (298 K).

Isosteric heats of adsorption (Q_{st})

The isosteric heats of adsorption (Q_{st}) for all the gases were fitted by a virial method (1) using the adsorption isotherms at 273 K and 298 K, and then calculated by using the Clausius-Clapeyron equation (2).

$$\ln P = \ln N + 1/T \sum_{i=0}^{m} a_i N^i + \sum_{j=0}^{n} b_j N^j$$
(1)
$$Q_{st} = -R \sum_{i=0}^{m} a_i N^i$$
(2)

Where *P* is pressure (mmHg), *N* is the amount adsorbed quantity (mmol g^{-1}), *T* is the temperature (K), a_i and b_i are virial coefficients, *R* is the real gas constant, and *m*, *n* represent the number of coefficients required to adequately describe the isotherms.

Prediction of the gases adsorption selectivity by IAST

IAST (ideal adsorption solution theory) was used to predict binary mixture adsorption from the experimental pure-gas isotherms. For C_2H_2 and CO_2 isotherms of the activated ST-**sod**-Co/Ti in the low pressure, the experimental isotherm data (measured at 273 and 298 K) were fitted using the single-site Langmuir-Freundlich equation (3).

$$N = A \frac{B \times P^C}{1 + B \times P^C}$$
(3)

Where, N is molar loading of species i (mmol g^{-1}), A is saturation capacity of species i (mmol g^{-1}), B is Langmuir constant (kPa^{-C}), C is Freundlich constant, P is bulk gas phase pressure of species i (kPa) The adsorption selectivity (*S*_{ads}) for binary mixture of C₂H₂/CO₂, defined by

$$S_{ads} = \frac{x_1 / x_2}{y_1 / y_2}$$
(4)

Where x_i is the mole fractions of component i in the adsorbed phases and y_i is the mole fractions of component i in the bulk phases.

Column Breakthrough Experiments

The mixed-gas breakthrough separation experiment was carried out using a home-built setup coupled with a mass spectrometer (Pfeiffer GSD320). In a typical breakthrough experiment for C_2H_2/CO_2 (50:50, v/v) gas mixtures, powder (0.45 g) was packed into a custom-made stainless-steel column (3.0 mm I.D.×120 mm) with silica wool filling the void space. The packed column was heated at 100 °C for 12 h under a constant He flow (10 mL min⁻¹ at 298 K and 1 bar) to activated the sample. The flow of He was then turned off and a gas mixture of C_2H_2/CO_2 (50:50, v/v) (1 mL min⁻¹) was allowed to flow into the column. Outlet effluent from the column was continuously monitored using mass spectrometer. After the breakthrough experiment, the sample was regenerated in-situ in the column at 100 °C for 12 h.

Additional Figures and Descriptions



Fig. S1. The one-pot synthesis of the ST-sod-MOFs by mixed-linkers tpt and btc with homometallic or heterometallic trimers $[M_3(OH/O)(COO)_6]$. Cages are illustrated with light pink and green balls.



Fig. S2. A view of 3D porous framework of ST-sod-MOFs along [001] (left) and [111] (right) directions, respectively.



Fig. S3. Two types of polyhedral cages with different sizes observed in ST-sod-MOFs.



Fig. S4. The 3D framework of ST-sod-MOFs constructed from trimer clusters, btc and tpt ligands corresponding linkage between nodes and topological net with the Schäfli symbol of $(4^3)_6(4^6 \cdot 6^{15} \cdot 8^7)_3(6^3)_2$ topology.

Topological analysis results for ST-sod-MOFs, the trimer clusters and the btc and tpt ligands reduced

into 8-, 3-, 3-connected nodes, respectively:

Structure consists of 3D framework with V3Ti2Sc6 Vertex symbols for selected sublattice

Sc1 Point symbol: {4^3} Extended point symbol: [4.4.4]

Ti1 Point symbol: {6^3}

Extended point symbol:[6(4).6(4).6(4)]

V1 Point symbol: {4^6.6^15.8^7}

Extended point symbol:

Point symbol for net: {4^3}6{4^6.6^15.8^7}3{6^3}2

3,3,8-c net with stoichiometry (3-c)6(3-c)2(8-c)3; 3-nodal net



Fig. S5. The 3D framework of ST-sod-MOFs reveal the underlying sod net formed by the 4-c

supertetrahedron SBBs.

Structure consists of 3D framework with Sc

Vertex symbols for selected sublattice

Sc1 Point symbol: $\{4^2.6^4\}$

Extended point symbol: [4.4.6.6.6.6]

Point symbol for net: $\{4^2.6^4\}$

4-c net; uninodal net

Topological type: sod/SOD; 4/4/c1; sqc970 (topos&RCSR.ttd) {4^2.6^4} - VS [4.4.6.6.6.6]

Physical Measurements



Fig. S6. The SEM image (inset) and EDX analysis of ST-sod-MOFs.



Fig. S7. The Co2p spectrum of ST-**sod**-Co. The satellite structure and the energetic positions of the $Co2p_{3/2}$ (781.0 eV) and $Co2p_{1/2}$ (796.8 eV) peaks indicate divalent high-spin states of the Co ions in

ST-sod-Co.



Fig. S8. The Ni2p spectrum of ST-**sod**-Ni. The satellite structure and the energetic positions of the Ni $2p_{3/2}$ (855.8 eV) and Ni $2p_{1/2}$ (873.4 eV) peaks show divalent high-spin states of the Co ions in ST-

sod-Ni.



Fig. S9. The Co2p (left) and Ti2p (right) spectra of ST-sod-Co/Ti. The satellite structure and the energetic positions of the $Co2p_{3/2}$ (781.8 eV) and $Co2p_{1/2}$ (797.7 eV) peaks are characteristic of the Co^{2+} ions in ST-sod-Co/Ti. Two peaks located at around 458.8 eV and 465.1 eV, correspond to $Ti2p_{3/2}$ and $Ti2p_{1/2}$, respectively, indicating an oxidation state of the Ti^{4+} ions in ST-sod-Co/Ti.



Fig. S10. Experimental and simulated PXRD patterns for different samples.



Fig. S11. Experimental and simulated PXRD patterns for different samples.





Fig. S12. In situ variable temperature PXRD patterns for ST-sod-Ni and ST-sod-Co/Ti under room atmosphere.



Fig. S13. Experimental and simulated PXRD patterns for ST-sod-MOFs.



Fig. S14. The PXRD patterns for ST-sod-Co and ST-sod-Ni after being soaked in water in 12 h and 24

h at 25 $^{\circ}$ C.



Fig. S15. The TGA curves of ST-**sod**-Co, ST-**sod**-Ni and ST-**sod**-Co/Ti. The TGA curves revealed that ST-**sod**-Co, ST-**sod**-Ni, and ST-**sod**-Co/Ti undergo successive weight losses in the range 30-200 °C, 30-200 °C, and 30-210 °C, respectively, corresponding to the release of the coordinated waters and guest molecules. And the frameworks begin to decompose at ~ 320 °C for ST-**sod**-Co, ~ 350 °C for

ST-sod-Ni, ~ 380 °C for ST-sod-Co/Ti, respectively.



Fig. S16. N_2 adsorption isotherm at 77 K and BET surface area of ST-sod-Co.



Fig. S17. N_2 adsorption isotherms at 77 K and BET surface area of ST-sod-Ni.



Fig. S18. N₂ adsorption isotherms at 77 K and BET surface area of ST-sod-Co/Ti.



Fig. S19. Single-component adsorption (filled circles) and desorption (open circles) isotherms of different gases measured at 273 K (left) and 298 K (right) for ST-sod-Co.



Fig. S20. Single-component adsorption (filled circles) and desorption (open circles) isotherms of different gases measured at 273 K (left) and 298 K (right) for ST-sod-Ni.



Fig. S21. Single-component adsorption (filled circles) and desorption (open circles) isotherms of different gases measured at 273 K (left) and 298 K (right) for ST-sod-Co/Ti.



Fig. S22. The parameters and optimized adsorption isotherms for the isosteric heats of adsorption (Q_{st}) of C_2H_2 for ST-sod-Co calculated by the Clausius–Clapeyron equation.



Fig. S23. The parameters and optimized adsorption isotherms for the isosteric heats of adsorption (Q_{st}) of CO₂ for ST-sod-Co calculated by the Clausius–Clapeyron equation.



Fig. S24. The parameters and optimized adsorption isotherms for the isosteric heats of adsorption (Q_{st}) of C_2H_2 for ST-sod-Ni calculated by the Clausius–Clapeyron equation.



Fig. S25. The parameters and optimized adsorption isotherms for the isosteric heats of adsorption (Q_{st}) of CO₂ for ST-sod-Ni calculated by the Clausius–Clapeyron equation.



Fig. S26. The parameters and optimized adsorption isotherms for the isosteric heats of adsorption (Q_{st}) of C_2H_2 for ST-sod-Co/Ti calculated by the Clausius–Clapeyron equation.



Fig. S27. The parameters and optimized adsorption isotherms for the isosteric heats of adsorption (Q_{st}) of CO₂ for ST-sod-Co/Ti calculated by the Clausius–Clapeyron equation.



Fig. S28. A comparison of the isosteric heat at zero-loading for C₂H₂ and CO₂ on ST-sod-MOFs.

Identification code	ST-sod-Co	ST-sod-Ni	ST-sod-Co/Ti
CCDC	2026913	2026914	2026915
Empirical formula	$C_{30}H_{17}Co_{3}N_{4}O_{14}$	$C_{30}H_{17}Ni_{3}N_{4}O_{14}$	$C_{30}H_{17}Co_2TiN_4O_{14}$
Formula weight	834.26	833.30	823.24
Temperature/K	100(4)	104.5(7)	105(2)
Crystal system	cubic	cubic	cubic
Space group	Im 3 m	Im 3 m	Im 3 m
a=b=c/Å	33.2828(3)	32.97540(10)	33.2244(2)
$\alpha = \beta = \gamma / ^{\circ}$	90	90	90
Volume/Å ³	36868.9(10)	35856.7(3)	36675.1(7)
Z	24	24	24
$ ho_{calc} g/cm^3$	0.899	0.830	0.889
µ/mm ⁻¹	6.630	5.431	3.826
F(000)	9960.0	9010.0	9800.0
Crystal size/mm ³	0.10×0.10×0.10	0.129×0.129×0.129	0.16×0.12 ×0.10
Radiation	CuK α (λ = 1.54184)	GaKa ($\lambda = 1.34050$)	GaKa ($\lambda = 1.34050$)
2Θ range for data collection/°	6.506 to 152.688	5.708 to 121.466	7.316 to 122.018
Index ranges	$\begin{array}{l} -28 \leq h \leq 41, \\ -27 \leq k \leq 41, \\ -23 \leq l \leq 28 \end{array}$	$\begin{array}{l} -42 \leq h \leq 42, \\ -31 \leq k \leq 42, \\ -38 \leq l \leq 42 \end{array}$	$\begin{array}{l} -24 \leq h \leq 43, \\ -22 \leq k \leq 43, \\ -40 \leq l \leq 43 \end{array}$
Reflections collected	28342	100150	80009
Independent reflections	3601 [$R_{int} = 0.0592$, $R_{sigma} = 0.0369$]	$3885 [R_{int} = 0.0524, R_{sigma} = 0.0135]$	$3954 [R_{int} = 0.0493, R_{sigma} = 0.0158]$
Data/restraints/parameters	3601/24/133	3885/20/135	3954/60/164
Goodness-of-fit on F^2	1.055	1.038	1.058
Final R indexes [I>= 2σ (I)]	$R_1 = 0.0759,$ $wR_2 = 0.2216$	$R_1 = 0.0629,$ $wR_2 = 0.1774$	$R_1 = 0.0536,$ $wR_2 = 0.1623$
Final R indexes [all data]	$R_1 = 0.0983,$ $wR_2 = 0.2401$	$R_1 = 0.0656,$ $wR_2 = 0.1802$	$R_1 = 0.0590,$ $wR_2 = 0.1679$
Largest diff. peak/hole / e Å ⁻³	1.54/-0.69	1.30/-0.63	0.91/-0.83

Table S1. Crystal Data and Structure Refinements for ST-sod-MOFs.

Material	BET Surface Area m ² /g	Pore Volume cm ³ /g	Pore Size Å	Pore Aperture Å	Topology Type	Ref.
ST-sod-Co/Ti	2362	0.71	22	5.60	sod	This work
ST-sod-Ni	1783	0.62	22	5.51	sod	This work
ST- sod -Co	1767	0.58	22	5.57	sod	This work
ZIF-8	1630	0.64	11.6	3.4	sod	[1]
MIL-100(Fe)	1456	1.25	25, 29	5.5, 8.6	mtn	[2]
sph-MOF-4	2170	1.14	22	/	sph	[3]
MOF-808	2060	0.84	18.4	/	spn	[4]
Al-CAU-42	2170	0.81	16	/	β- cristobalite	[5]
PCN-777	2008	2.82	35	/	β- cristobalite	[6]
MOF-500	2274	0.61	9.6/14.8/20	3.4/6.4/9.5	β- cristobalite	[7]
MOF-818	2050	/	37	/	spn	[8]

 Table S2. Summary of Gas Adsorption Properties for ST-sod-MOFs and ZIF-8 and

 Selected Supertetrahedron-Based MOFs.

Material	BET Surface Area m ² /g	Pore Volume cm ³ /g	C2H2 Qst kJ/mol	CO ₂ Q _{st} kJ/mol	Ref.
ST-sod-Co/Ti	2362	0.71	42.4	32.6	This work
ST-sod-Ni	1783	0.62	25.2	31.1	This work
ST-sod-Co	1310	0.62	29.1	32.1	This work
JCM- 1	550	/	36.9	33.4	[9]
DICRO- 4- Ni- i	398	0.22	37.7	33.9	[10]
UTSA- 74a	830	0.39	31	25	[11]
TCuCl	167	/	41	30.1	[12]
NKMOF-1-Ni	382	/	60.3	40.9	[13]
UTSA-300a	311	0.16	57.6	/	[14]

Table S3. Comparison of Heat of Adsorption Data for C_2H_2 and CO_2 in Various MOFs.

Material	BET Surface Area m ² /g	Pore Volume cm ³ /g	C ₂ H ₂ /CO ₂ selectivity value	Ref.
ST-sod-Co/Ti	2362	0.71	1.65	This work
Zn-MOF-74	/	/	2.0	[11]
FJU-90a	1572	0.65	4.3	[15]
TIFSIX-2-Cu-i	685	0.33	6.5	[16]
UTSA-74a	830	0.39	9.0	[11]

Table S4. Comparison of Adsorption Selectivity Value of Equimolar BinaryC2H2/CO2 Gas Mixtures in Selected Metal-organic Frameworks at 1.0 Bar and 298 K.

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