# Supporting Information for

# Metal-Hydrogen-Pi-Bonded Organic Frameworks

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## Contents

Section 1. Materials and Methods	2
Section 2. Ligand Synthesis and Characterizations	5
Section 3. Scanning Electron Microscopy of M-HAF-2	7
Section 4. Structural characterization of M-HAF-2	7
Section 5. Gas adsorption, Thermogravimetric, PXRD and Zeta Potential Analyses	11
Section 6. Dye uptake/Ionic Separation of Fe-HAF-2	14
Section 7. Postsynthetic Metal Exchange of M-HAF-2	
Reference	25

#### **Section 1. Materials and Methods**

All chemicals were purchased from commercial sources and used without further purification unless otherwise mentioned.

Synthesis of Fe-HAF-2. FeCl<sub>3</sub>·6H<sub>2</sub>O (8.1 mg, 0.03 mmol) and H<sub>4</sub>TPTH (13.98 mg, 0.03 mmol) were added in a mixed solution of containing 1.8 mL *N*,*N*-diethylformamide (DEF) and 0.2 mL acetonitrile (ACN) (v:v = 9:1) in a 1-dram vial and sonicated for 10 min. Then, the solution was heated in a 60 °C oven for 48 h. After cooling to 25 °C, the resultant solid was isolated by centrifugation, washed with fresh DEF (3×1 mL), and acetone (3×1 mL). Orange crystalline powders were obtained (7 mg, 32% yield).

Synthesis of Ga-HAF-2. The stock solution of GaCl<sub>3</sub> in DEF (100 mg/mL) was prepared because GaCl<sub>3</sub> is hygroscopic in air. 53  $\mu$ L of GaCl<sub>3</sub> (5.28 mg, 0.03 mmol) stock solution and H<sub>4</sub>TPTH (9.33 mg, 0.02 mmol) were added in a mixed solution of containing 1.4 mL DEF and 0.6 mL acetonitrile (v:v = 7:3) in a 1-dram vial and sonicated for 10 min. Then, the solution was heated in a 100 °C oven for 48 h. After cooling to 25 °C, the resultant solid was isolated by centrifugation, washed with fresh DEF (3×1 mL), and acetone (3×1 mL). Ivory crystalline powders were obtained (6 mg, 40% yield).

Synthesis of In-HAF-2. InBr<sub>3</sub> (21.2 mg, 0.06 mmol) and H<sub>4</sub>TPTH (13.98 mg, 0.03 mmol) were added in a mixed solution of 1 mL DEF and 1 mL acetonitrile (v:v = 1:1) in a 1-dram vial and sonicated for 10 min. Then, the solution was heated in a 100 °C oven for 48 h. After cooling to 25 °C, the resultant solid was isolated by centrifugation, washed with fresh DEF (3×1 mL), and acetone (3×1 mL). White crystalline powders were obtained (7 mg, 30% yield).

**Single Crystal Electron Diffraction Analysis.** Continuous rotation electron diffraction (cRED) data were collected under cryogenic temperature (98 K) on a 200 kV JEOL JEM-2100-LaB<sub>6</sub>

transmission electron microscope (Cs 1.0 mm, point resolution 0.23 nm) equipped with a hybrid Timepix detector (resolution 512×512 pixels, pixel size 55  $\mu$ m) from Amsterdam Scientific Instruments. Images were recorded with a Gatan Orius 833 CCD camera (resolution 2048×2048 pixels, pixel size 7.4  $\mu$ m) under low dose conditions.

**X-Ray Diffraction Analyses.** Powder X-ray diffraction (PXRD) patterns were measured at room temperature in transmission mode on a Bruker SMART Pt135 CCD diffractometer equipped with Cu K $\alpha$  radiation ( $\lambda$  =1.54178 Å) calibrated with AgBeH. Images were stitched together and azimuthally averaged using DIFFRAC.EVA. The dry powder samples were mounted on cryoloops with Paratone oil.

Sample Activation and N<sub>2</sub> Gas Sorption Analysis. Before a gas sorption experiment, assynthesized M-HAF-2 (~50 mg) samples were washed with DEF three times and acetone three times, followed by soaking in acetone for 3–5 days to allow solvent exchange. During the solvent exchange process, the acetone was replaced with fresh solvent at least 3 times every 24 h. The resulting exchanged samples were evacuated in a vacuum oven overnight at 100 °C prior to analysis. Then the samples were degassed at 120 °C on a Micromeritics ASAP 2020 Adsorption Analyzer for a minimum of 12 h prior to gas adsorption/desorption measurements. Sorption data and Brunauer-Emmett-Teller (BET) surface area (m<sup>2</sup>/g) measurements were collected at 77 K with N<sub>2</sub> on a Micromeritics ASAP 2020 Adsorption Analyzer using volumetric technique.

**Thermogravimetric Analysis (TGA).** 5 mg of sample was placed in a 100 μL aluminum crucible. Samples were analyzed on a Mettler Toledo Star TGA/DSC using a temperature range of 30–500 °C scanning at 5 °C/min synthetic air (75 cm<sup>3</sup>/min air flow rate) for sample degradation measurements and a heat-cool-heat procedure at 10 °C/min for melting point determination.

**Stability Tests.** Chemical stability of each sample was determined by adding a small amount of freshly synthesized M-HAF-2 (5–10 mg) into different vials containing 2 mL of solvent. The MOF powders were soaked for 1 week after which they were isolated by centrifugation, washed with acetone three times, and dried under vacuum at room temperature. The resultant samples were analyzed by PXRD and compared to the pristine MOFs. Thermal stability of M-HAF-2 was assessed by *in situ* variable temperature PXRD experiments. A small amount of MOF powders was added into a 1-mm glass capillary (Hampton Research). The measurements were performed using a Bruker Microstar APEX II CCD diffractometer equipped with Cu K $\alpha$  radiation ( $\lambda$  =1.54178 Å).

**Scanning electron microscopy imaging (SEM).** The dry MOF powders were suspended in ethanol and deposited onto silicon chips with 5×5 mm dimensions on top of conductive carbon tape. SEM images were obtained using a Zeiss Sigma 500 scanning electron microscope (Zeiss) at an accelerating voltage of 1 kV to 1.5 kV using a 30-µm aperture with ETD detector.

**Zeta Potential Measurements.** Approximately 1 mg of M-HAF-2 (M = Fe, Ga, In) powders were suspended in 1 mL of aqueous solution at different pH values. The zeta potentials of M-HAF-2 MOFs in different pH solutions were determined using a Zetasizer Nano ZS90 (Malvern Instruments). Experimental runs were performed using an automatic collection mode.

**Dye uptake of Fe-HAF-2.** In a typical experimental set-up for dye uptake study, 5 mg of Fe-HAF-2 were dispersed in 20 mL of 10 ppm aqueous dye solutions. Absorption of the supernatant solution was monitored by UV-vis absorption spectroscopy over the time.

**Postsynthetic Cation Exchange of M-HAF-2.** 5 mg of as-synthesized M-HAF-2 and selected metal salts were added to 2 mL DEF in a 1-dram vial and sonicated for 10 min. Then, the solution

was heated in an oven at 80 °C for 48 h. After cooling to 25 °C, the resultant solid was isolated by centrifugation, washed with fresh DEF (3×1 mL), and acetone (3×1 mL).

Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The MOF samples were digested in 70% nitric acid and heated at 80 °C for 12 h. After filtration through a syringe filter (<0.2  $\mu$ m), the solution was diluted with water so that the final concentration of nitric acid was 0.7% by volume. All samples were analyzed for iron (Fe), gallium (Ga) and indium (In) content using a NexION 2000 ICP-MS. The reported values are the average of triplicate values. The calibration curve was established using a standard solution with a dwell time of 50 ms, thirty sweeps, and three replicates with background correction.

## Section 2. Ligand Synthesis and Characterizations

**1.1** Synthesis of 3,3",5,5"-tetramethyl-1,1':4',1"-terphenyl (H<sub>4</sub>TPTM)

3,3'',5,5''-tetramethyl-1,1':4',1''-terphenyl was synthesized as previously described with minor modifications.<sup>1</sup> 1,4-dibromobenzene (2.52 g, 10.68 mmol), 3,5-dimethylphenylboronic acid (4 g, 26.7 mmol) and Cs<sub>2</sub>CO<sub>3</sub> (9.326 g, 28.6 mmol) were combined in EtOH (70 mL), and the mixture was de-aerated using N<sub>2</sub>. Pd(PPh<sub>3</sub>)<sub>4</sub> (0.123 g, 0.107 mmol) was added, and the reaction mixture was refluxed at 100 °C for 48 h under N<sub>2</sub>. The reaction mixture was evaporated to dryness, and the residue extracted into CHCl<sub>3</sub> and washed with water. The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed *in vacuo*. The crude product was purified by silica gel chromatography using 5% Ethyl Acetate in hexane as the eluent. The product was obtained as a colorless crystalline solid. Yield: 2.5 g, 81.5%. <sup>1</sup>HNMR: (300 MHz, DMSO-d<sup>6</sup>)  $\delta$  7.71(s, 4H),  $\delta$  7.31 (s, 4H),  $\delta$  7.01 (s, 2H),  $\delta$  2.34 (s, 12H).

## **1.2** Synthesis of [1,1':4',1"] terphenyl-3,3",5,5"-tetracarboxylic acid (H<sub>4</sub>TPTC)

[1,1':4',1"]terphenyl-3,3",5,5"-tetracarboxylic acid was synthesized as previously described with minor modifications.<sup>1</sup> 3,3",5,5"-tetramethyl-p-terphenyl (3 g, 10.47 mmol) and KMnO<sub>4</sub> (6.74 g, 42.6 mmol) was dissolved in 150 mL of pyridine and 150 mL of H<sub>2</sub>O. The reaction was heated at 100 °C, and KMnO<sub>4</sub> (5 g x 6 portions) was added over 3 days. The reaction mixture was cooled and filtered over Celite. The filtrate was evaporated to remove ssolvent and dissolved in 50 mL H<sub>2</sub>O. The mixture as filtered again and the filtrate was acidified using concentrated HCl until no additional white precipitate formed (pH = 2). The white precipitate was filtered, washed with water and then with MeOH, and dried in vacuo.

Yield: 3.5 g, 82.1%. <sup>1</sup>HNMR: (300 MHz, DMSO-d<sup>6</sup>) δ 8.49 (t, 2H), δ 8.46 (d, 4H), δ 7.92 (s, 4H),

### **1.3** Synthesis of [1,1':4',1"] terphenyl-3,3",5,5"-tetrahydroxamic acid (H<sub>4</sub>TPTH)

*O*-tritylhydroxylamine was synthesized as previously described.<sup>2</sup> [1,1':4',1"]terphenyl-3,3",5,5"-tetracarboxylic acid (2.13 g, 5.24 mmol), HATU (8.77 g, 23.06 mmol), DIPEA (10.99 mL, 62.9 mmol), and *O*-tritylhydroxylamine (6.93 g, 25.2 mmol) were added to 75 mL of DMF and stirred overnight at room temperature. The solution was added to 100 mL of ethyl acetate and extracted with H<sub>2</sub>O (3 x 50 mL). The precipitate (4.52 g, 3.14 mmol) was suspended in 20 ml dichloromethane with 10% (v/v) trifluoroacetic acid and stirred for 30 min at room temperature. The remaining pre-cipitate was filtered, washed with 20 mL dichloromethane, and dried *in vacuo*. White powder (1.40 g, 3.01 mmol, 58% yield) was obtained as final product. <sup>1</sup>HNMR: (300 MHz, DMSO-d<sup>6</sup>)  $\delta$  11.45 (s, 4H),  $\delta$  9.19 (s, 4H),  $\delta$  8.21 (s, 4H),  $\delta$  8.17 (s, 2H),  $\delta$  7.95 (s, 4H).



Scheme S1. Synthetic scheme for the H<sub>4</sub>TPTH ligand.



Fig. S1. <sup>1</sup>HNMR of H<sub>4</sub>TPTH in DMSO-d<sub>6</sub>.

Section 3. Scanning Electron Microscopy of M-HAF-2



Fig. S2. SEM images for Fe-HAF-2.

# Section 4. Structural characterization of M-HAF-2

TEM and cRED sample preparation was performed by crashing the powder of Fe-HAF-2 with pestle and mortar and dispersing it in acetone. Three droplets of the resulting dispersion were transferred onto a carbon-coated copper grid. To avoid the damage from high vacuum, the grid was loaded on a single-tilt cryo-sample holder (tilting range  $-60^{\circ}$  to  $+60^{\circ}$ ) and the data was collected under cryogenic temperature (98 K). For data collection, a crystal was placed in the

electron beam and brought to the mechanical eucentric height. The goniometer was continuously rotated while selected-area electron diffraction patterns were simultaneously captured from the crystal. The cRED data were collected using the software *Instamatic*<sup>3</sup> with camera length of 30 mm, exposure time of 0.5 s per frame, and rotation speed of goniometer of  $0.45^{\circ}$  s<sup>-1</sup>.

The space group and unit cell parameters were initially obtained using the software  $REDp^4$ . From the two-dimensional (2D) slices cut from the reconstructed 3D reciprocal lattice (Fig. S3) the reflection conditions can be deduced as  $h\bar{h}l$ : l=2n; 00l: l=2n, which suggests the possible space groups of P3c1 (No. 158),  $P\bar{3}c1$  (No. 165),  $P6_3cm$  (No. 185),  $P\bar{6}c2$  (No. 188), and  $P6_3/mcm$  (No. 193). The data were subsequently processed by XDS<sup>5</sup>. The unit cell parameters determined from cRED data were, a = 18.200(3) Å, and c = 16.170(3) Å. Pawley fit was applied to further confirm and refine the unit cell parameters, which converged to a = 18.263(2), and c = 16.177(2), with  $R_p$ = 0.0095,  $R_{wp} = 0.0130$ ,  $R_{exp} = 0.0174$ , and GOF = 0.751 (Table S1).

Two datasets from individual crystals with the highest resolution were selected and merged by XSCALE, which is part of XDS package, for structural determination. The structure was solved *ab initio* using the space group  $P\bar{3}c1$  by dual-space algorithm implemented in SHELXT. Notably, atomic scattering factors for electrons were applied. All the atom positions could be directly located from the initial structure solution. Only the nitrogen atoms were wrongly assigned the atom type, and they were corrected according to the chemical information of the linkers.

The final full-matrix least square refinement was performed by SHELXL<sup>6, 7</sup>, using kinematical intensity assumption. Soft geometrical restrains (DFIX, DANG) were applied on the linker molecules to soften the distortions of the benzene rings. Restrains for isotropic displacement parameters (EADP) were applied for the C atoms in the benzene rings. At the end of the refinement, the SWAT<sup>8</sup> parameter was applied to compensate the effects of residual solvents in the pores. The crystallographic data and structure refinement details are given in Table S2.



**Fig. S3.** (a) The reconstructed 3D reciprocal lattice of Fe-HAF-2 viewed along the  $c^*$ -axis. 2D slices show the (b)  $h\bar{h}l$ , and (c) *hhl* planes. (d) The crystal from which the cRED data were collected.

Crystal system	Trigonal
Space group	<i>P</i> 3 <i>c</i> 1 (No. 165)
<i>a</i> , <i>c</i> (Å)	18.263(2), 16.177(2)
Temperature (K)	298(2)
Wavelength (Å)	1.54056
$2\theta$ range (°)	3.00 - 54.99
$\mathbf{R}_p$	0.0095
$R_{wp}$	0.0130
R <sub>exp</sub>	0.0174
GOF	0.751

 Table S1. Crystallographic data and Pawley fit results of Fe-HAF-2.

	2 (// 0.020111)
Number of merged datasets	2
Chemical formula	$FeC_{33}N_6O_{12}$
Formula weight	728.13
Ζ	4
Crystal system	Trigonal
Space group	<i>P</i> 3 <i>c</i> 1 (No. 165)
<i>a</i> , <i>c</i> (Å)	18.200(3), 16.170(3)
Temperature (K)	98
Resolution (Å)	1.10
Number of total reflections	8841
Number of total unique reflections	1634
Number unique reflections (I > 2 sigma(I))	612
Number of parameters	111
Number of restrains	22
Completeness(%)	93.6
Rint	0.2565
R <sub>1</sub> for Fo>4 sigma (Fo)	0.2092
$R_1$ for all	0.3285
Goof	1.324

**Table S2.** Crystallographic details of Fe-HAF-2 ( $\lambda = 0.0251$  Å)



**Fig. S4**. Pawley fitting of powder X-ray diffraction for Fe-HAF-2. Red line: calculated; black line: observed; blue line: difference, grey bars: Bragg positions.

Section 5. Gas adsorption, Thermogravimetric, PXRD and Zeta Potential Analyses



Fig. S5. N<sub>2</sub> adsorption/desorption isotherms of In-HAF-2 measured at 77 K.



Fig. S6. TGA analysis of M-HAF-2.



Fig. S7. In situ variable temperature PXRD of Ga-HAF-2 (a) and In-HAF-2 (b).



**Fig. S8.** PXRD patterns of Ga-HAF-2 (a) and In-HAF-2 (b) after incubation in aqueous solutions at different pH values.



Fig. S9. Zeta potential of M-HAF-2 as a function of pH.





Fig. S10. Molecular dimensions of dyes with different charges and the pore size of Fe-HAF-2.



**Fig. S11.** UV-vis spectra of an aqueous solution of LV<sup>+</sup> in the presence of Fe-HAF-2 collected at various time points.



**Fig. S12.** UV-vis spectra of an aqueous solution of a)  $OG^{2-}$  and b)  $AO6^{-}$  in the presence of Fe-HAF-2 collected at various time points.



**Fig. S13.** UV-vis spectra of an aqueous solution of NR in the presence of Fe-HAF-2 collected at various time points.



**Fig. S14.** PXRD pattern of Fe-HAF-2 after dye uptake experiments compared with the assynthesized sample.

### Pseudo-second-order kinetic model

The kinetics of  $LV^+$  and  $MB^+$  adsorption for different initial dye concentrations can be described by the pseudo-second-order kinetic model, using the equation 1:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$

with

$$h = k_2 q_e^2$$

where h is the initial sorption rate; t is the adsorption time (s),  $q_e$  and  $q_t$  are the amount adsorbed at equilibrium and at time t, respectively (mg·g<sup>-1</sup>), and  $k_2$  is the rate constant for pseudo-secondorder adsorption (g·mg<sup>-1</sup>·s<sup>-1</sup>).  $k_2 = \frac{slope^2}{intercept}$  when the  $\frac{t}{q_t}$  is plotted against t.



Fig. S15. Plot of pseudo-second-order kinetics for the uptake of MB<sup>+</sup> by Fe-HAF-2.

Table S2 Kinetic parameters for the adsorption of MB<sup>+</sup> on Fe-HAF-2 at room temperature.

Dye	C <sub>0</sub>	pН	Removal	Pseudo-second-order kinetic model			
	(ppm)		(%)	qe	$k_2$ (g·mg <sup>-</sup>	h (mg·g-	<b>R</b> <sup>2</sup>
				(mg/g)	$^{1} \cdot s^{-1}$ )	$^{1} \cdot s^{-1}$ )	
$MB^+$	10	6.97	99	40.16	4.38×10 <sup>-3</sup>	7.064	0.9999
	20	6.61	99	80.62	7.405×10 <sup>-4</sup>	4.813	0.9999
	40	6.77	94	155.77	7.746×10 <sup>-5</sup>	1.879	0.9977

Section 7. Postsynthetic Metal Exchange of M-HAF-2



Fig. S16. PXRD patterns of M-HAF-2 after post-synthetic modification with additional FeCl<sub>3</sub>



**Fig. S17.** a) The reconstructed 3D reciprocal lattice of Ga-HAF-2 + FeCl<sub>3</sub> viewed along the  $c^*$ -axis. The inset is the TEM image of the crystal from which the dataset was collected. 2D slices show the b)  $h\bar{h}l$ , and c) *hhl* planes. The structural model viewed along the d) [001], e) [010], f) [110] directions. g) The structure model highlighting the uncoordinated hydroxamic acid groups.



Fig. S18.  $N_2$  adsorption/desorption isotherms of In-HAF-2 after PSE with GaCl<sub>3</sub> measured at 77 K.



**Fig. S19.** a) PXRD patterns of In-HAF-2 as synthesized, In/Fe-1, In/Fe-2, In/Fe-3 and In/Fe-4. b) Photos of In-HAF-2 and In/Fe-4. c) SEM images of In-HAF-2 and d) In/Fe-1 to 4.



Fig. S20.  $N_2$  adsorption/desorption isotherms of In-HAF-2 after PSE with FeCl<sub>3</sub> measured at 77 K.

**Table S3.** Extents of transmetalation calculated based on the ICP-MS of digested In-HAF-2samples after PSE upon the addition of different amounts of FeCl3.

Sample	Metal ratio in reaction	Normalized metal ratio after digestion for ICP-MS	Empirical formula of MOF based on ICP-MS	Extent of transmetalation in In- HAF-2
In/Fe-1	In:Fe = 1:1	1:1.5	In <sub>0.80</sub> Fe <sub>1.20</sub> (TPTH) <sub>3</sub>	60%
In/Fe-2	In:Fe = $1:2.5$	1:3.2	In <sub>0.48</sub> Fe <sub>1.52</sub> (TPTH) <sub>3</sub>	76%
In/Fe-3	In : Fe = 1:5	1:7.7	In <sub>0.23</sub> Fe <sub>1.77</sub> (TPTH) <sub>3</sub>	89%
In/Fe-4	In : Fe = $1:10$	1:8.2	$In_{0.22}Fe_{1.78}$ (TPTH) <sub>3</sub>	89%



**Fig. S21.** a) PXRD patterns of Ga-HAF-2 as synthesized, Ga/Fe-1, Ga/Fe-2, Ga/Fe-3 and Ga/Fe-4. b) Photos of Ga-HAF-2 and Ga/Fe-4. c) SEM images of Ga-HAF-2 and d) Ga/Fe-1 to 4.

**Table S4.** Extent of transmetalation calculated based on the ICP-MS of digested Ga-HAF-2 samples after PSE upon the addition of different amounts of FeCl<sub>3</sub>.

Sample	Metal ratio in	Normalized metal	Empirical	Extent of
	reaction	ratio after	formula of MOF	transmetalation in
		digestion for ICP-	based on ICP-	Ga-HAF-2
		MS	MS	
Ga/Fe-1	Ga : Fe = 1: 1	1: 3.8	$Ga_{0.42}Fe_{1.58}$	79.0%
			(TPTH) <sub>3</sub>	
Ga/Fe-2	Ga : Fe = 1: 2.5	1: 5.3	Ga <sub>0.16</sub> Fe <sub>1.74</sub>	87.0%
			(TPTH) <sub>3</sub>	
Ga/Fe-3	Ga : Fe = 1: 5	1: 7.4	Ga <sub>0.12</sub> Fe <sub>1.88</sub>	94.0%
			(TPTH) <sub>3</sub>	
Ga/Fe-4	Ga : Fe = 1: 10	1: 7.7	Ga <sub>0.11</sub> Fe <sub>1.89</sub>	94.5%
			(TPTH) <sub>3</sub>	

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