# Supporting Information

# Tuning the Moisture Stability of Metal-Organic Frameworks by Incorporating Hydrophobic Functional Groups at Different Positions of Ligands

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# Experimental

## 1. Preparation

All materials and solvents were obtained from commercial suppliers (Sigma-Aldrich, Alfa Aesar, and others) and used without further purification.

*1.1. Synthesis of $P^2$ and $P^3$ ligands*

The preparations of 2,2'-dimethyl-4,4'-bipyridine ($P^2$) and 3,3'-dimethyl-4,4'-bipyridine ($P^3$) were carried out according to the reported procedures.[1]

*1.2. Synthesis of MOF-508·(DMF)·$(H_2O)_2$ ($[Zn_2(BDC)_2(P^1)]$·(DMF)·$(H_2O)_2$)*

A mixture of $Zn(NO_3)_2·6H_2O$ (89.1 mg, 0.3 mmol), $H_2BDC$ (52.8 mg, 0.3 mmol), 4,4'-bipyridine (23.4 mg, 0.15 mmol) was suspended in DMF (15 mL) and heated to 120 °C for 48 h in a 23 mL Teflon-lined stainless-steel autoclave, followed by cooling to room temperature at 2 °C $h^{-1}$ to yield colorless block-shaped single crystals of MOF-508·(DMF)·$(H_2O)_2$ (yield: 50.1%, based on Zn). Elemental analysis (%) calcd for $C_{29}H_{27}Zn_2N_3O_{11}$ (723.0), C, 48.13; H, 3.73; N, 5.81. Found: C, 48.11; H, 3.77; N, 5.78. Main IR frequencies (KBr, $cm^{-1}$): 3386(s) (O-H (water)), 1654(m) (C=O (DMF)), 1579(vs), 1502(w), 1383(vs), 1190(w), 1148(w), 1110(w), 1081(w), 1013(w), 883(w), 853(w), 828(w), 745(s), 652(w), 572(w), 513(w).

*1.3. Synthesis of SCUTC-18·$(DMF)_2$·$(H_2O)$ ($[Zn_2(BDC)_2(P^2)]$·$(DMF)_2$·$(H_2O)$)*

SCUTC-18 was prepared by using the same procedures as those for MOF-508 except that $P^1$ was replaced by 2,2'-dimethyl-4,4'-bipyridine (27.6 mg, 0.15 mmol). Yield 55.4% (based on Zn). Elemental analysis (%) calcd for $C_{34}H_{36}Zn_2N_4O_{11}$ (806.4), C, 50.62; H, 4.47; N, 6.95. Found: C, 50.58; H, 4.50; N, 6.96. Main IR frequencies (KBr, $cm^{-1}$): 3438(s) (O-H (water)), 2935(w) ($C_{methyl}$-H of $P^2$), 1659(m) (C=O (DMF)), 1578(s), 1503(w), 1389(s), 1089(m), 1150(w), 1100(w), 1058(w), 1015(w), 887(w), 823(m), 748(s), 667(w), 545(w).

*1.4. Synthesis of SCUTC-19·(DMF)·$(H_2O)_2$ ($[Zn_2(BDC)_2(P^3)]$·(DMF)·$(H_2O)_2$)*

SCUTC-19 was prepared by using the same procedure as those for MOF-508 except that $P^1$ was replaced by 3,3'-dimethyl-4,4'-bipyridine (27.6 mg, 0.15 mmol). Yield 53.4% (based on Zn). Elemental analysis (%) calcd for $C_{31}H_{31}Zn_2N_3O_{11}$ (751.0), C, 49.53; H, 4.13; N, 5.59. Found: C, 49.50; H, 4.15; N, 5.61. Main IR frequencies (KBr, $cm^{-1}$): 3435(s) (O-H (water)), 2951(w) ($C_{methyl}$-H of $P^3$), 1657(m) (C=O (DMF)), 1578(vs), 1502(vs), 1387(s), 1150(w), 1108(w), 1015(w), 886(w), 818(w), 746(s), 661(w), 517(w).

*1.5. Preparation of fully desolvated MOF508, SCUTC-18 and SCUTC-19 ($[Zn_2(BDC)_2(P)]$, P = $P^1$, $P^2$, or $P^3$)*

The resultant crystalline material was immersed in DMF for 24 h and then sequentially in chloroform for three 24 h periods. Finally, the compound was dried under vacuum for 12 h at 120 °C to fully remove the solvent. Elemental analysis caldc (%) for $Zn_2(BDC)_2(P)$ (615.04 for MOF-508, 643.04 for SCUTC-18 and SCUTC-19): C 50.73%, H 2.60%, N 4.55% for MOF-508, C 52.25%, H 3.11%, N 4.55% for SCUTC-18 and SCUTC-19; found: C 50.76%, H 2.56%, N 4.56% for MOF-508, C 52.28%, H 3.13%, N 4.52% for SCUTC-18 and C 52.27%, H 3.11%, N 4.53% for SCUTC-19.

**2. Crystal structure determination**

Single crystal X-ray diffraction analyses of complexes MOF-508·(DMF)·$(H_2O)_2$, SCUTC-18·$(DMF)_2$·$(H_2O)$, and SCUTC-19·(DMF)·$(H_2O)_2$ were performed on a Rigaku Mercury CCD diffractometer operated at 90 kV and 50 mA using Mo Kα radiation (λ = 0.71073 Å) at room temperature. The empirical absorption corrections were performed using the CrystalClear program.[2] The structures were solved by direct methods and refined on $F^2$ by full-matrix least squares technique using the SHELX-97 program package.[3] All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms attached to carbon were placed in geometrically idealized positions and refined using a riding model. The routine SQUEEZE (PLATON)[4] was applied to the structures in order to remove

diffuse electron density associated with badly disordered water and DMF molecules. One of the 2-methyl-byridine ring of $P^2$ in SCUTC-18 is disordered and they are split into two sets of positions, with occupancy rations of 0.470(5):0.530(5). Due to the significant overlap of the disordered atoms the following restraints were applied: the ring (C22 C23 C24 C25 C26 N2 C27) and their disordered counterparts were each restrained to be flat and their equivalent bond distances were restrained to be the same within a standard deviation of 0.01 Å. The crystallographic data for SCUTC-18 and SCUTC-19 are given in Table S1. Selected bond lengths and bond angles are given in Table S2.

Crystallographic data for MOF-508: triclinic *P-1*, with a = 10.894(2) Å, b = 10.955(2) Å, c = 14.102(3) Å, α = 89.19(3)˚, β = 88.64(3)˚, γ = 81.33(3)˚, V = 1663.2(6) $Å^3$, Z = 2, μ(Mo Ka) = 1.482$mm^{-1}$, ρ = 1.228 g $cm^{-3}$, T = 293 K, reflection numbers collected = 13368, unique reflections (Rint) = 5959 (0.0530), $R_1$ [I>2\s(I)] = 0.0575 and $wR_2$ (all data) = 0.1443, GOF = 1.150.

Crystallographic data for SCUTC-18: Tetragonal $P4_32_12$, with a = 10.9288(4) Å, b = 10.9288(4) Å, c = 56.2967(2) Å, α =β = γ = 90 ˚, V = 6724.0(4) $Å^3$, Z = 4, μ(Mo Ka) = 1.484$mm^{-1}$, ρ = 1.410 g $cm^{-3}$, T = 293 K, reflection numbers collected = 12976, unique reflections (Rint) = 5626 (0.0437), $R_1$ [I>2\s(I)] = 0.0518 and $wR_2$ (all data) = 0.1726, GOF = 1.073, Flack = 0.34(3). CCDC: 806376.

Crystallographic data for SCUTC-19: triclinic *P-1*, with a = 10.925(2) Å, b = 10.946(2) Å, c = 13.972(3) Å, α = 95.19(3) ˚, β = 99.23(3) ˚, γ = 99.89(3) ˚, V = 1612.7(6) $Å^3$, Z = 2, μ(Mo Ka) = 1.532$mm^{-1}$, ρ = 1.325 g $cm^{-3}$, T = 293 K, reflection numbers collected = 12733, unique reflections (Rint) = 5736 (0.1001), $R_1$ [I>2\s(I)] = 0.0759 and $wR_2$ (all data) = 0.1807, GOF = 0.941. CCDC: 806377.

**3. Characterization**

Elemental analyses for C, H, and N were carried out by using a Vario EL III Elemental Analyzer. Infrared (IR) spectra were recorded (4000-400 $cm^{-1}$) as KBr disks on a Bruker 1600 FTIR spectrometer. Thermogravimetry analyses (TGA) were performed on a simultaneous SDT thermal analyzer (STA449C, Netzsch) under a flow of $N_2$ at a heating rate of 10 °C/min between ambient temperature and 800 °C. Powder XRD investigations were carried out on a Bruker AXS D8-Advanced diffractometer at 40 kV and 40 mA with Cu Kα (λ = 1.5406 Å) radiation.

**4. Sorption measurements**

The $N_2$ isotherm was measured with an automatic volumetric adsorption apparatus (Micrometrics ASAP 2020) at 77 K. The sorption isotherm for toluene vapor was measured with an automatic gravimetric adsorption apparatus (IGA-003 series, Hiden Isochema Ltd.) at 298 K. Prior to measurements, the desolvated sample was further treated under high vacuum at 393 K overnight. The kinetic trap effects of water (or toluene) on the MOFs were monitored by TGA. Before measurements, the sample was treated at 473 K in flowing $N_2$ overnight to remove the guest molecules. After cooling to room temperature, the sample was exposed to a $N_2$ flow with water (or toluene) vapor until no weight change was observed. Then the sample was heated at a rate of 5 °C/min under a pure $N_2$ flow.

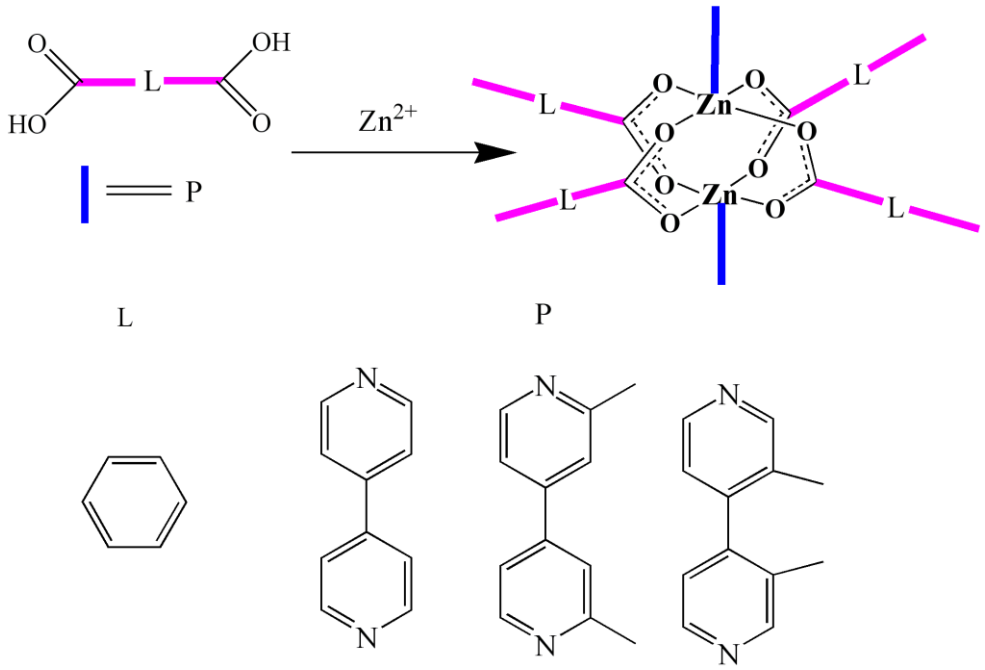
**References**

[1] P. Leighton, J. K. M. Sanders, *J. Chem. Soc. Perkin Trans. I* **1987**, 2385-2393.

[2] Molecular Structure Corporation and Rigaku, **2000**. CrystalClear. Version 1.36. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA, and Rigaku Corporation, 3-9-12 Akishima, Tokyo, Japan.

[3] G. M. Sheldrick, *Acta Cryst.* **2008**, *A64*, 112-122.

[4] A. L. Spek, *PLATON, A Multipurpose Crystallographic Tool*; Utrecht University: Utrecht, The Netherlands, **2005**.




**Scheme S1.** Schematic illustration of the self-assembly of paddle-wheel cluster $Zn_2(CO_2)_4$ with bicarboxylate $L(COO)_2$ and bidentate pillar linkers P to construct 3D primitive cubic MOFs, $Zn_2(L(COO)_2)_2(P)$.

**Table S1.** Crystal data and structure refinement details of SCUTC-18 and SCUTC-19.

| | SCUTC-18 | SCUTC-19 |
|---|---|---|
| Empirical Formula | $C_{34}H_{36}Zn_2N_4O_{11}$ | $C_{31}H_{31}Zn_2N_3O_{11}$ |
| Formula weight | 806.4 | 751.0 |
| Crystal System | Tetragonal | Triclinic |
| Space group | $P4_32_12$ | $P\text{-}1$ |
| $a$ (Å) | 10.9288(4) | 10.925(2) |
| $b$ (Å) | 10.9288(4) | 10.946(2) |
| $c$ (Å) | 56.2967(2) | 13.972(3) |
| $\alpha$ (°) | 90.00 | 95.20(3) |
| $\beta$ (°) | 90.00 | 99.23(3) |
| $\gamma$ (°) | 90.00 | 99.89(3) |
| $V$(Å$^3$) | 6724.0(4) | 1612.7(5) |
| $Z$ | 4 | 2 |
| $F$ (000) | 2597 | 652 |
| $D_{calc}$ (mg/cm$^3$) | 1.268 | 1.532 |
| Absorption coefficient (mm$^{-1}$) | 1.470 | 1.532 |
| GOF | 1.073 | 0.941 |
| Crystal size (mm) | 0.30 × 0.25 × 0.21 | 0.30 × 0.23 × 0.18 |
| $\theta$ range for data collection (°) | 3.01-25.20 | 3.03-25.20 |
| Limiting indices | $-6 \leqslant h \leqslant 12$ | $-11 \leqslant h \leqslant 13$ |
| | $-12 \leqslant k \leqslant 13$ | $-13 \leqslant k \leqslant 13$ |
| | $-67 \leqslant l \leqslant 67$ | $-16 \leqslant l \leqslant 16$ |
| Reflections collected / unique | 12976/5626 | 12731/5736 |
| Completeness to $\theta$ = 26.99 | 98.70% | 98.80% |
| Data/restraints/parameters | 5626/395 | 5736/363 |
| Final $R$ indices [ I > 2$\sigma$(I)] | $R_1$ = 0.0518, $wR_2$ = 0.1759 | $R_1$ = 0.0759, $wR_2$ = 0.2137 |
| $R$ indices (all data) | $R_1$ = 0.0570, $wR_2$ = 0.1726 | $R_1$ = 0.1354, $wR_2$ = 0.1807 |

$R = \sum(\|F_o\| - |F_c|)/\sum|F_o|$.
$wR = [\sum w(F_o^2 - F_c^2)^2/\sum w(F_o)^2]^{1/2}$.

**Table S2.** Selected bond distances (Å) and angles (°).

| | | | |
|---|---|---|---|
| **SCUTC-18** | | | |
| Zn(1)-O(3)$^{i}$ | 2.023(4) | Zn(2)-O(6) | 2.033(3) |
| Zn(1)-O(1) | 2.030(4) | Zn(2)-O(8)$^{ii}$ | 2.042(4) |
| Zn(1)-O(7)$^{ii}$ | 2.032(5) | Zn(2)-O(4)$^{i}$ | 2.050(4) |
| Zn(1)-O(5) | 2.059(3) | Zn(2)-O(2) | 2.061(3) |
| Zn(1)-N(2) | 2.050(2) | Zn(2)-N(1)$^{iii}$ | 2.084(4) |
| | | | |
| O(3)$^{i}$-Zn(1)- O(1) | 164.32(15) | O(3)$^{i}$-Zn(1)-O(1)$^{ii}$ | 88.32(2) |
| O(3)$^{i}$-Zn(1)-O(5) | 88.81(2) | O(7)$^{ii}$-Zn(1)-N(2) | 111.10(14) |
| N(2)-Zn(1)-O(5) | 98.04(13) | O(1)-Zn(1)-N(2) | 96.62(14) |
| O(6)-Zn(2)-O(8)$^{ii}$ | 162.88(14) | O(8)$^{ii}$-Zn(2)-O(4)$^{i}$ | 86.69(2) |
| O(6)-Zn(2)-O(2) | 88.95(2) | O(8)$^{ii}$-Zn(2)-O(2) | 87.69(16) |
| O(4)$^{i}$-Zn(2)-O(2) | 149.17(14) | O(6)-Zn(2)-N(1)$^{iii}$ | 96.11(15) |
| O(8)$^{ii}$-Zn(2)-N(1)$^{iii}$ | 100.99(15) | O(2)-Zn(2)-N(1)$^{iii}$ | 98.35(15) |
| **SCUTC-19** | | | |
| Zn(1)-N(1) | 2.039(6) | Zn(2)-N(2)$^{i}$ | 2.054(6) |
| Zn(1)-O(2) | 2.027(5) | Zn(2)-O(1) | 2.034(6) |
| Zn(1)-O(6) | 2.083(6) | Zn(2)-O(5) | 2.023(5) |
| Zn(1)-O(8)$^{i}$ | 2.053(5) | Zn(2)-O(4) | 2.010(5) |
| Zn(1)-O(3)$^{iv}$ | 2.027(6) | Zn(2)-O(7)$^{i}$ | 2.062(5) |
| | | | |
| N(1)-Zn(1)-O(2) | 101.0(3) | N(1)-Zn(1)-O(3)$^{iv}$ | 97.5(3) |
| O(2)-Zn(1)-O(3)$^{iv}$ | 161.2(2) | O(2)-Zn(1)-O(8)$^{i}$ | 90.1(2) |
| O(3)$^{iv}$-Zn(1)-O(8)$^{i}$ | 87.1(2) | O(2)-Zn(1)-O(6) | 87.2(2) |
| O(3)$^{iv}$-Zn(1)-O(6) | 88.0(2) | O(8)$^{i}$-Zn(1)-O(6) | 156.7(2) |
| O(4)$^{iv}$-Zn(2)-O(5) | 90.9(2) | O(4)$^{iv}$-Zn(2)-N(2)$^{v}$ | 106.2(3) |
| O(4)$^{iv}$-Zn(2)-O(1) | 157.1(2) | O(5)-Zn(2)-O(1) | 86.9(2) |
| N(2)$^{v}$-Zn(2)-O(1) | 96.5(3) | O(4)$^{iv}$-Zn(2)-O(7)$^{i}$ | 86.8(2) |
| O(5)-Zn(2)-O(7)$^{i}$ | 162.1(2) | O(1)-Zn(2)-O(7)$^{i}$ | 88.4(2) |

Symmetry codes: i = -1+x, y, z; ii = x, -1+y, z; iii = 0.5-y, -0.5+x, -0.25+z; iv = x, 1+y, z; v = x, y, 1+z.

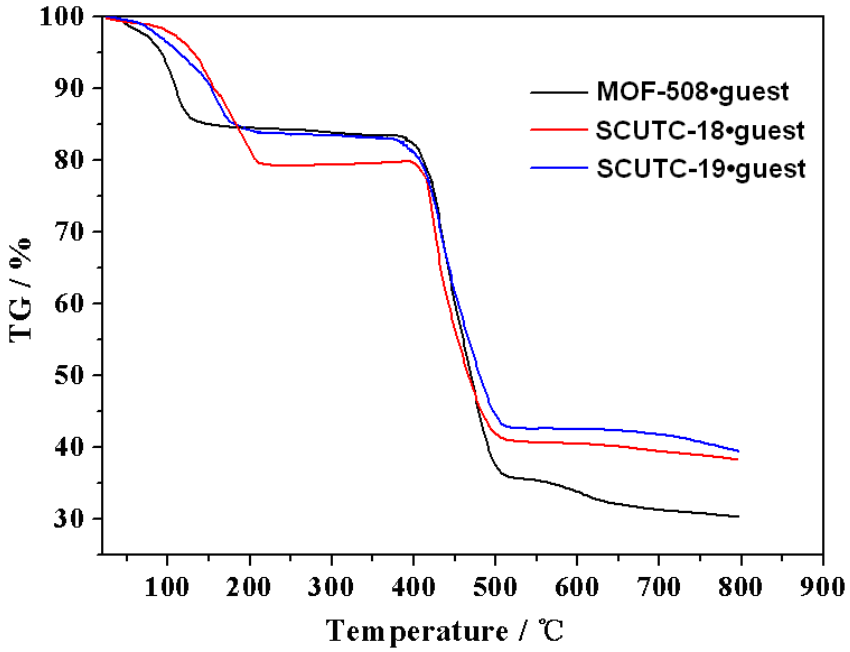



**Figure S1.** TGA curves for the as-synthesized compounds. MOF-508·(DMF)·$(H_2O)_2$: calcd weight loss (%) for DMF + $2H_2O$: 15.1; found: 15.0. SCUTC-18·$(DMF)_2$·$(H_2O)$: calcd weight loss (%) for 2DMF + $H_2O$: 20.4; found: 21.0. SCUTC-19·(DMF)·$(H_2O)_2$: calcd weight loss (%) for DMF + $2H_2O$: 14.6; found: 15.1.

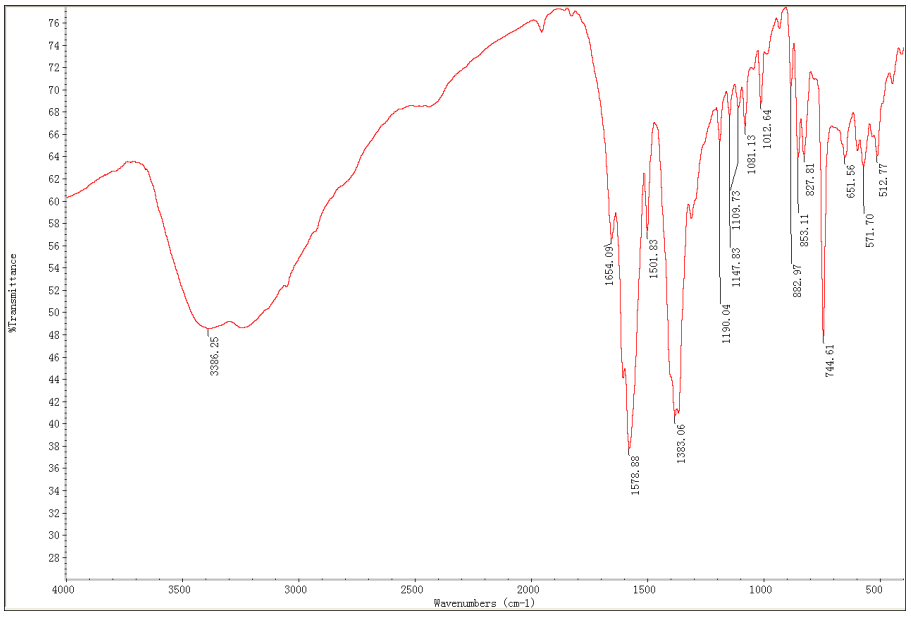



**Figure S2.** IR spectra of MOF-508·(DMF)·$(H_2O)_2$: 3386 $cm^{-1}$ ($O\text{-}H_{water}$), 1654 $cm^{-1}$ ($C{=}O_{DMF}$).

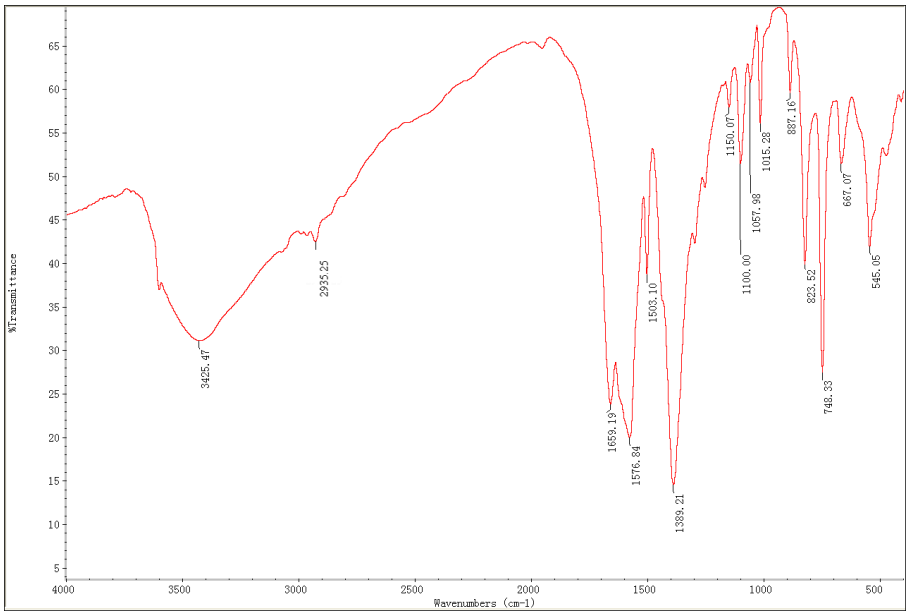



**Figure S3.** IR spectra of SCUTC-18·$(DMF)_2$·$(H_2O)$: 3425 $cm^{-1}$ ($O\text{-}H_{water}$), 1659 $cm^{-1}$ ($C{=}O_{DMF}$), 2935 $cm^{-1}$ ($C_{methyl}$-H of $P^2$).

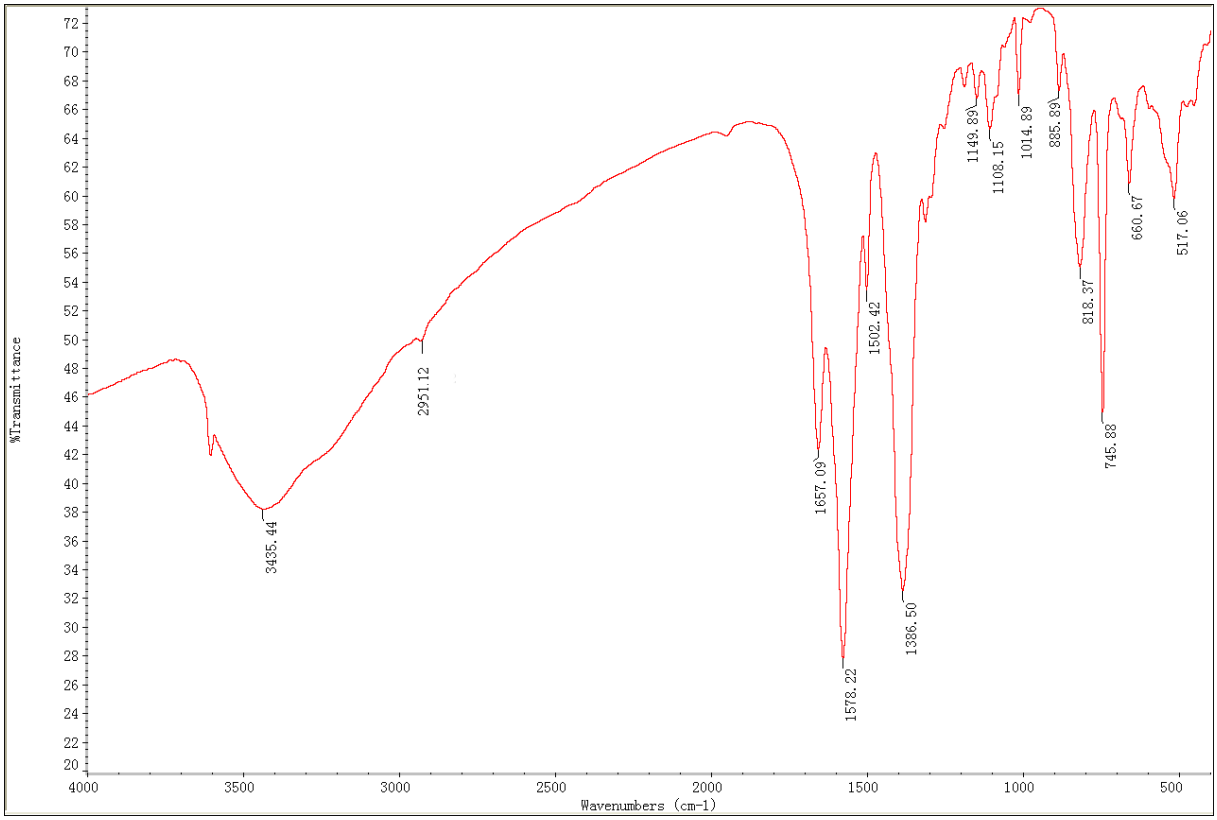



**Figure S4.** IR spectra of SCUTC-19·(DMF)·$(H_2O)_2$: 3435 $cm^{-1}$ ($O\text{-}H_{water}$), 1657 $cm^{-1}$ ($C{=}O_{DMF}$), 2951 $cm^{-1}$ ($C_{methyl}$-H of $P^3$).

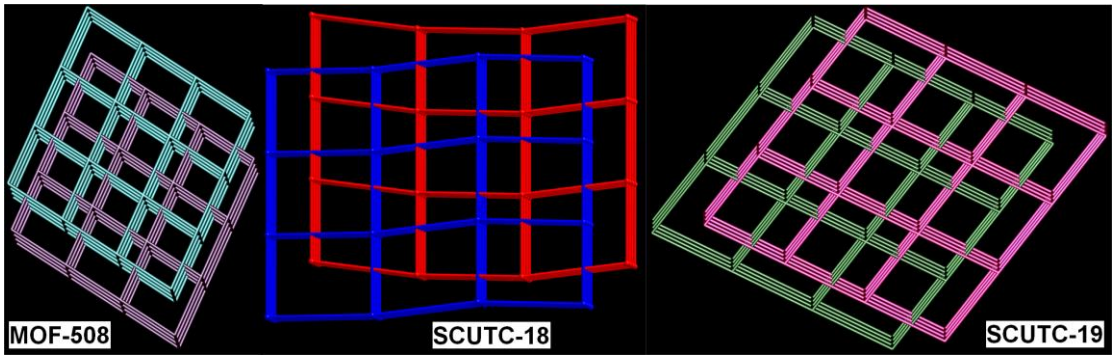



**Figure S5.** Showing the 2-fold interpenetration and remaining channels along the *c* axis.

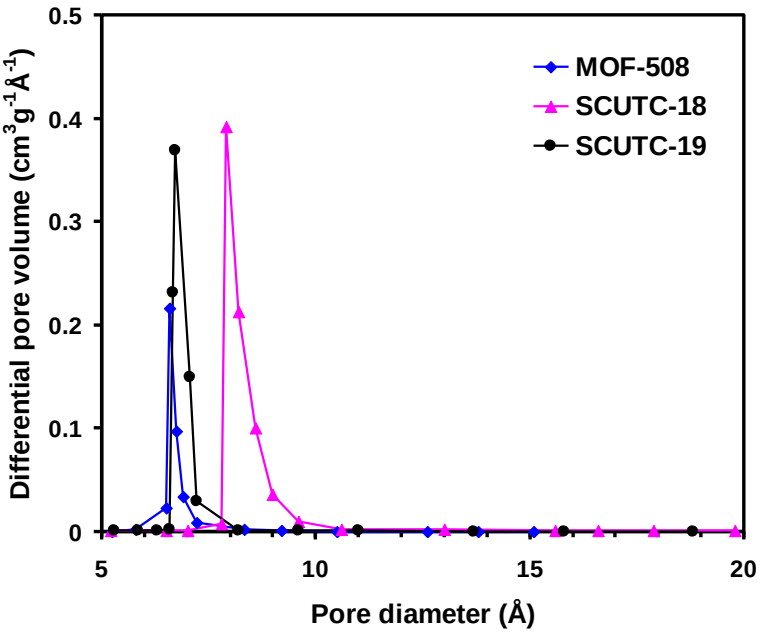
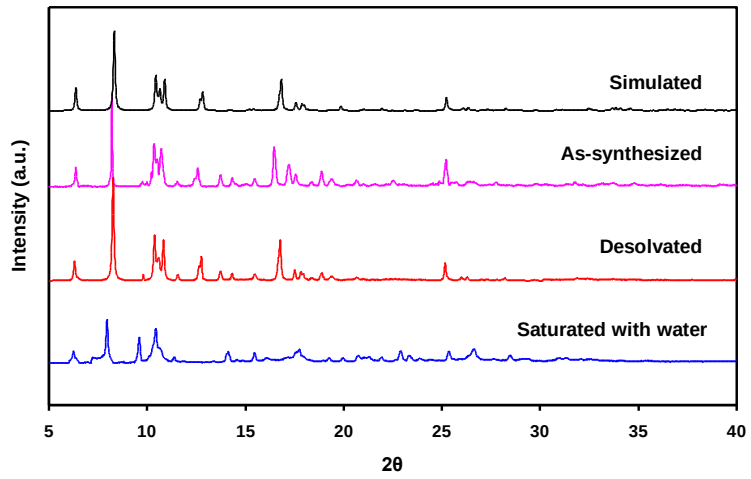
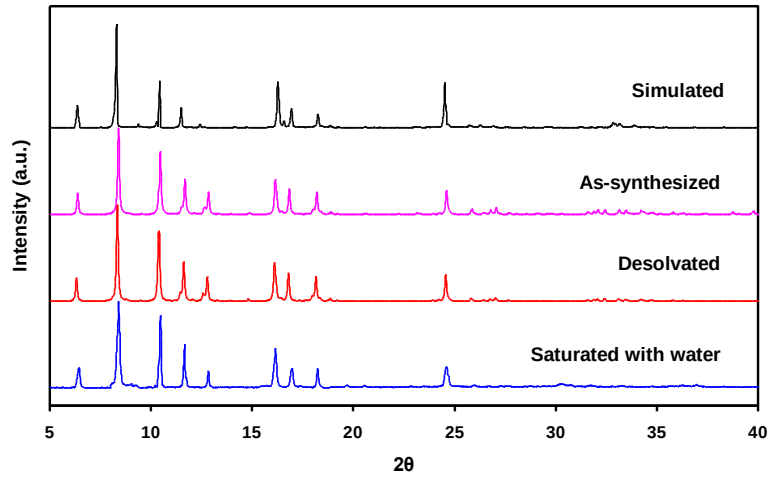



**Figure S6.** Micropore size distributions of MOF-508, SCUTC-18, and SCUTC-19.

(a)




(b)




(c)

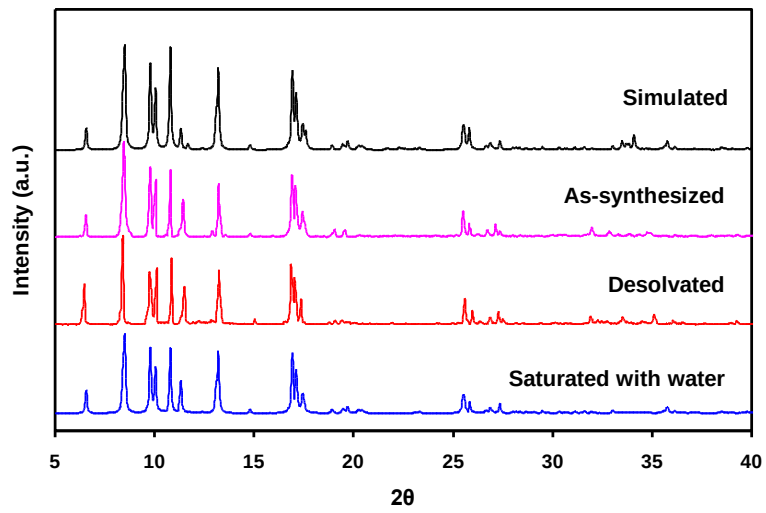



**Figure S7.** PXRD patterns of MOF-508 (a), SCUTC-18 (b), and SCUTC-19 (c). The desolvated samples were obtained by degassing the MOFs under vacuum at 473 K overnight. The MOFs saturated with water were obtained by using the same procedures for preparing the samples for TGA measurements (see Section 4 of the Experimental part of the ESI).

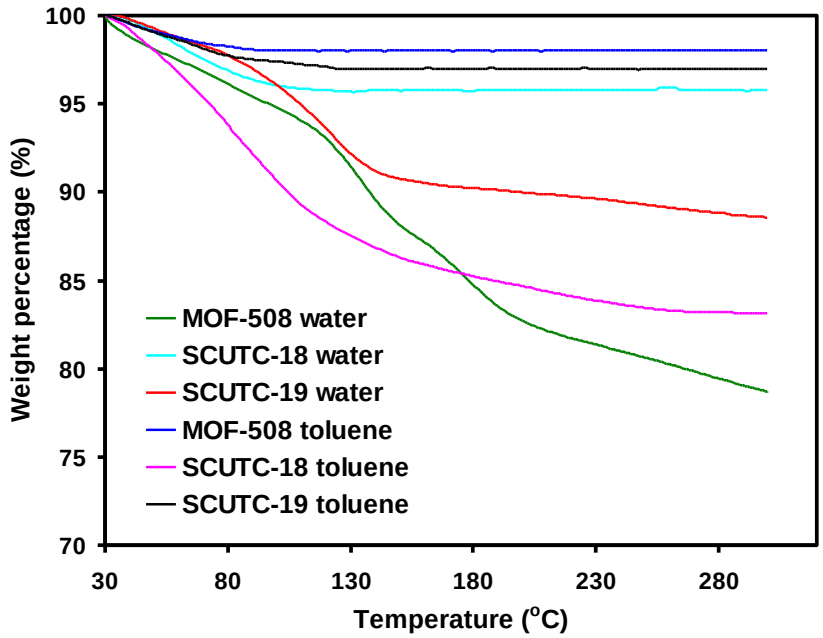



**Figure S8.** TGA of MOF-508, SCUTC-18, and SCUTC-19 with different absorbates.

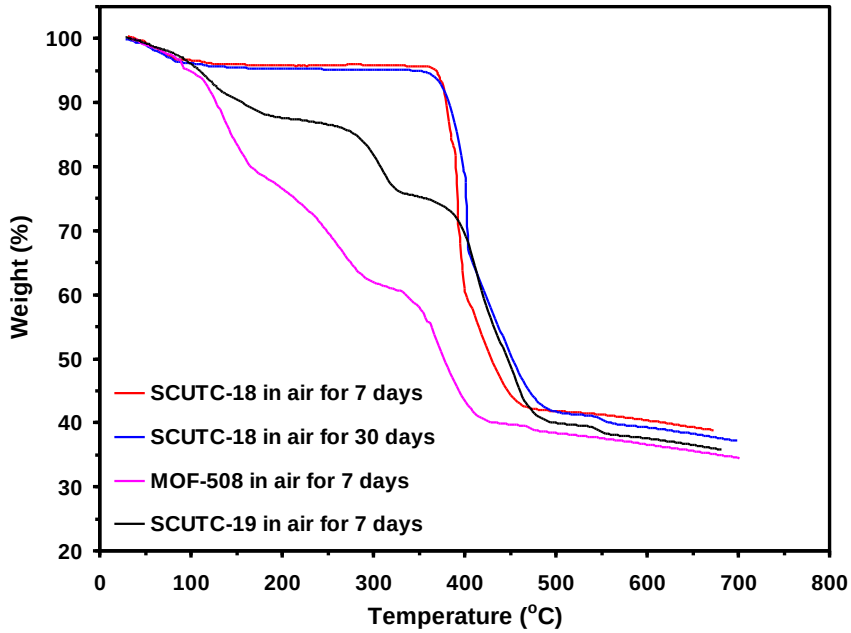



**Figure S9.** TGA of desolvated MOF-508, SCUTC-18, and SCUTC-19 after exposing to air for 7 or 30 days.

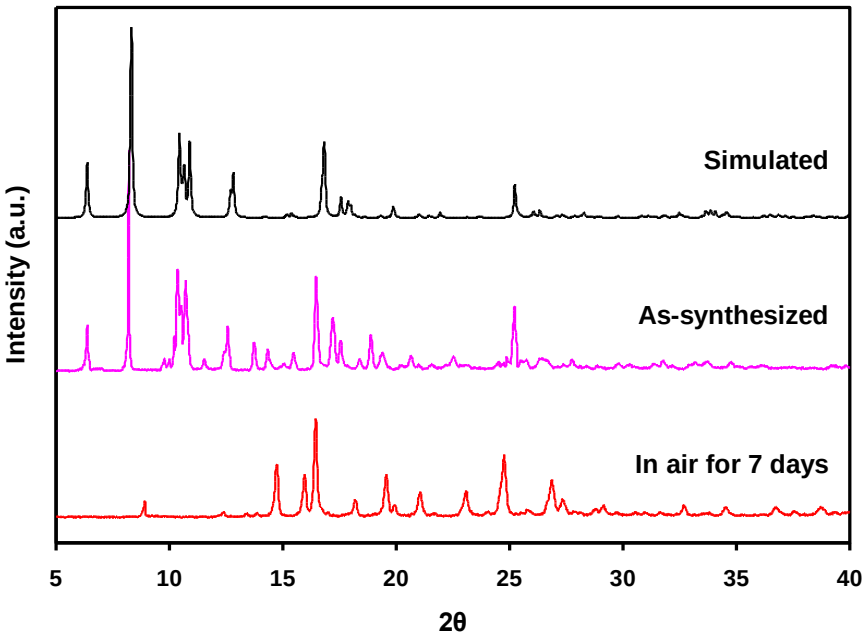



**Figure S10.** PXRD patterns of MOF-508 over a period of 7 days.

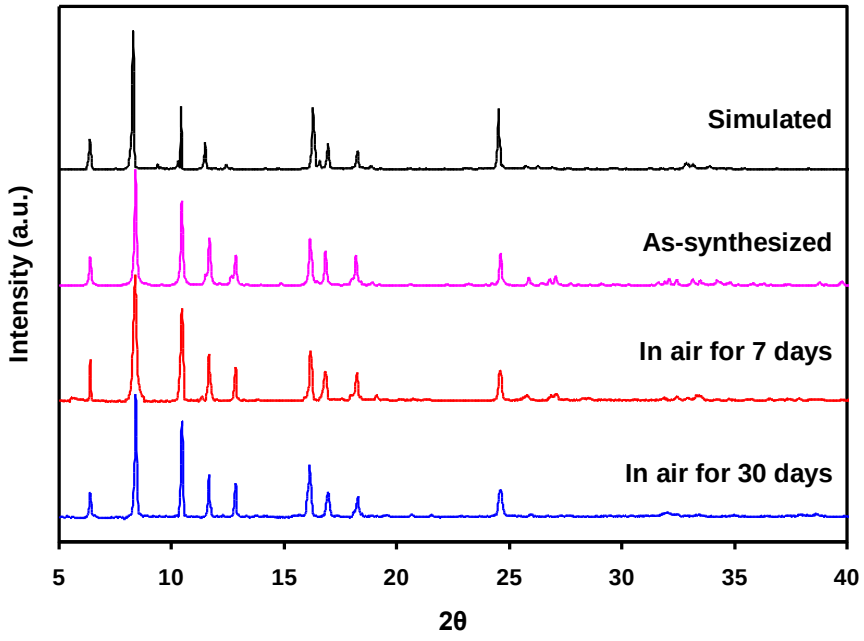



**Figure S11.** PXRD patterns of SCUTC-18 over a period of 30 days.

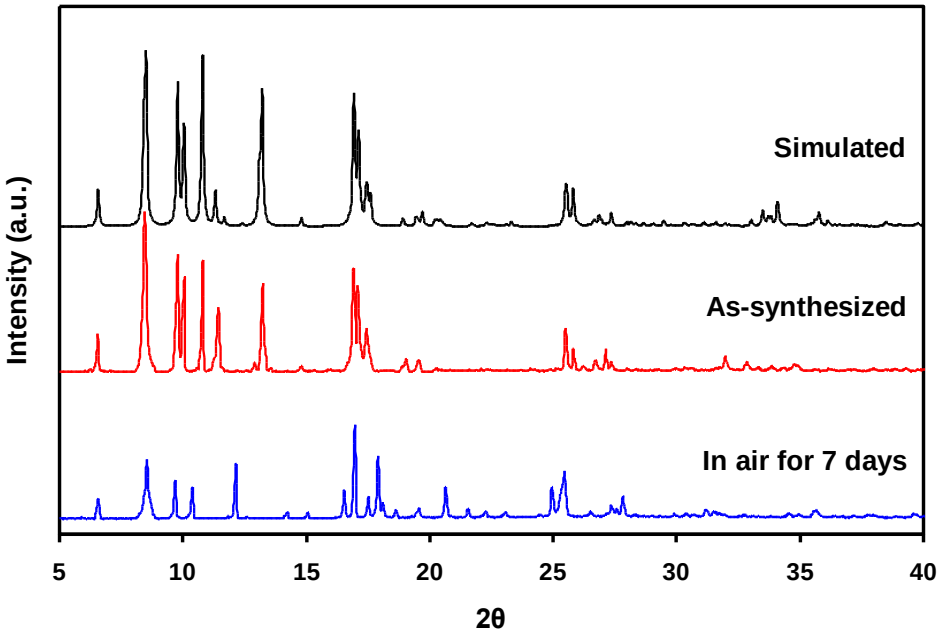



**Figure S12.** PXRD patterns of SCUTC-19 over a period of 7 days.

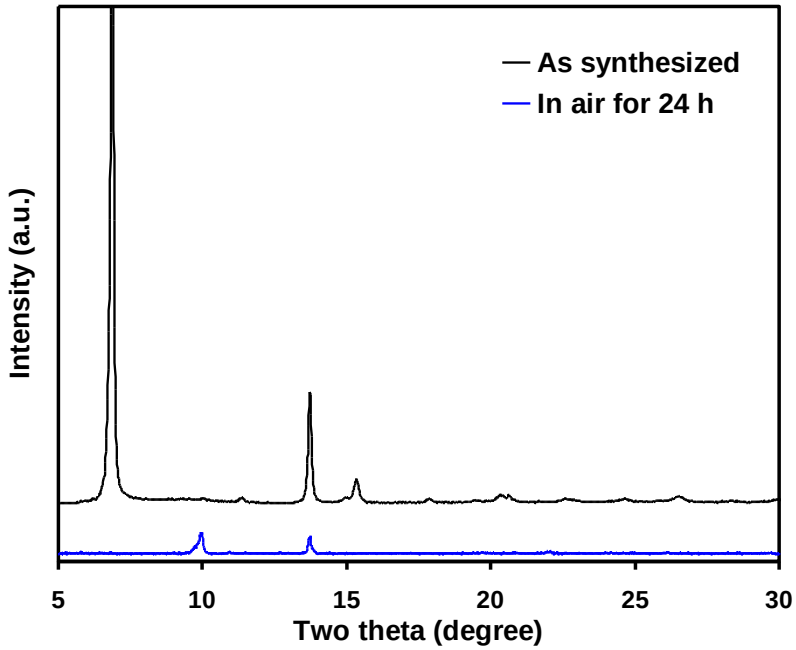



**Figure S13.** PXRD patterns of MTV-MOF-5-AF over a period of 24 h.

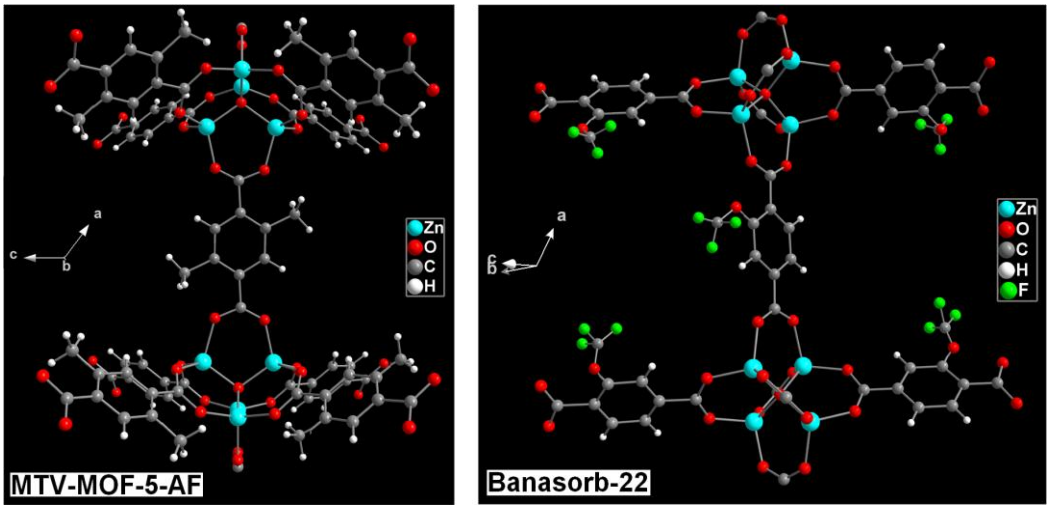



**Figure S14.** Views of the structures of MTV-MOF-5-AF and Banasorb-22.

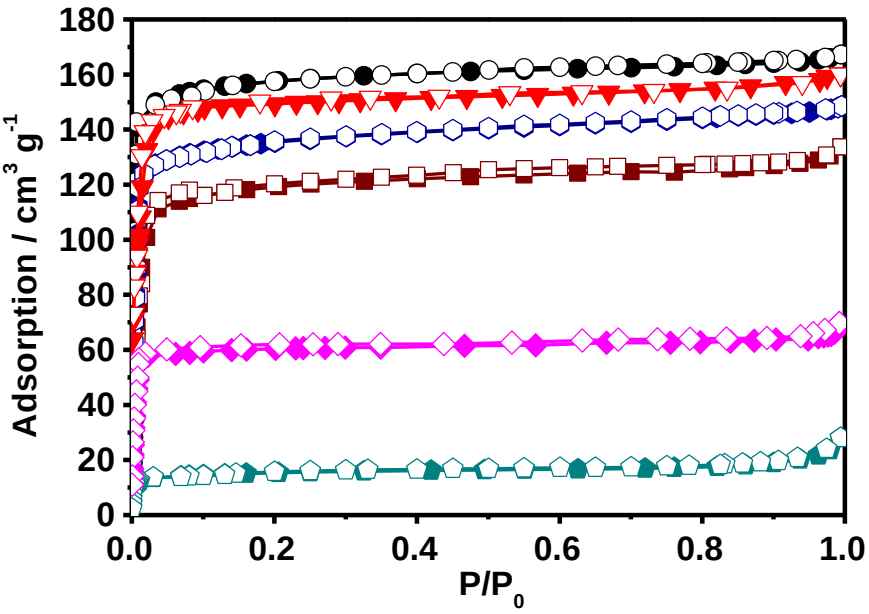



**Figure S15.** $N_2$ isotherms for MOF-508, SCUTC-18, and SCUTC-19 before and after exposing to air. Circle: SCUTC-18 as synthesized; triangle: SCUTC-18 exposed to air; hexagon: SCUTC-19 as synthesized; square: MOF-508 as synthesized; rhombus: SCUTC-19 exposed to air; pentagon: MOF-508 exposed to air. Filled symbols: adsorption; open symbols: desorption.

**Calculation of isosteric heat of adsorption**

The micropore filling of toluene vapor can be well described by the Dudinin-Radushkevich (DR) equation:

$$\ln W = \ln W_0 - (RT/\beta E_0)^2(\ln(P_0/P))^2$$

in which $W$ and $W_0$ are the amount of adsorption at $P/P_0$ and the saturated amount of adsorption, respectively. $\beta E_0$ is the adsorption energy.

The DR plot has a linear relationship in the high relative pressure ($P/P_0$) region, from which the value of $\beta E_0$ can be deduced (Figure S14). Furthermore, the isosteric heat of adsorption, $q_{st,\Phi=1/e}$, at the fractional filling of 1/e can be obtained by using $q_{st,\Phi=1/e} = \Delta H_v + \beta E_0$, where $\Delta H_v$ is the heat of vaporization of the solvent.

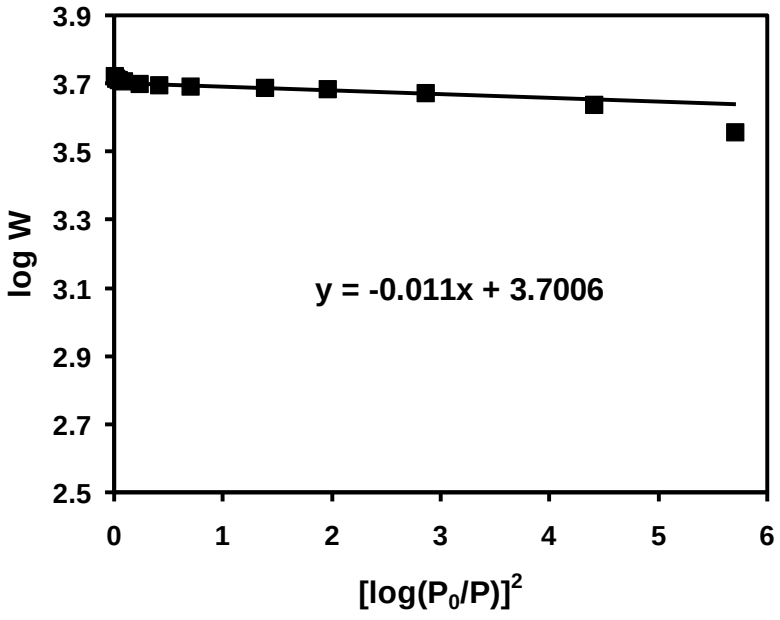



**Figure S16.** DR plot of toluene adsorption isotherm for SCUTC-18.

# checkCIF/PLATON report (basic structural check)

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report

## Datablock: SCUTC-18

Bond precision: C-C = 0.0070 A Wavelength=0.71073

Cell: a=10.9288(4) b=10.9288(4) c=56.2966(17)
alpha=90 beta=90 gamma=90

Temperature: 293 K

| | Calculated | Reported |
|---|---|---|
| Volume | 6723.99(10) | 6724.0(4) |
| Space group | P 43 21 2 | P 43 21 2 |
| Hall group | P 4nw 2abw | P 4nw 2abw |
| Moiety formula | C28 H18.59 N2 O8 Zn2 | C28 H18.59 N2 O8 Zn2 |
| Sum formula | C28 H18.59 N2 O8 Zn2 | C28 H18.59 N2 O8 Zn2 |
| Mr | 641.82 | 641.78 |
| Dx,g cm-3 | 1.268 | 1.268 |
| Z | 8 | 8 |
| Mu (mm-1) | 1.470 | 1.470 |
| F000 | 2596.7 | 2597.0 |
| F000' | 2602.34 | |
| h,k,lmax | 13,13,67 | 12,13,67 |
| Nref | 3631[ 6066] | 5626 |
| Tmin,Tmax | 0.650,0.734 | 0.662,0.746 |
| Tmin' | 0.637 | |

Correction method= MULTI-SCAN

Data completeness= 1.55/0.93 Theta(max)= 25.200

R(reflections)= 0.0518( 4933) wR2(reflections)= 0.1759( 5626)

S = 1.073 Npar= 395

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

## Alert level A

PLAT602_ALERT_2_A VERY LARGE Solvent Accessible VOID(S) in Structure !

---

## Alert level C

STRVA01_ALERT_4_C Flack test results are ambiguous.
From the CIF: _refine_ls_abs_structure_Flack 0.340
From the CIF: _refine_ls_abs_structure_Flack_su 0.030
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for O1
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for O5
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for O6
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for Zn1
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for Zn2
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds (x 1000) Ang .. 7
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond C17 - C18 ... 1.39 Ang.
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond C17 - C21 ... 1.37 Ang.
PLAT033_ALERT_4_C Flack x Parameter Value Deviates from Zero ..... 0.34
PLAT068_ALERT_1_C Reported F000 Differs from Calcd (or Missing)... ?
PLAT152_ALERT_1_C The Supplied and Calc. Volume s.u. Differ by ... - 30 Units

---

## Alert level G

REFLT03_ALERT_4_G Please check that the estimate of the number of Friedel pairs is
correct. If it is not, please give the correct count in the _publ_section_exptl_refinement section of the submitted CIF.
From the CIF: _diffrn_reflns_theta_max 25.20
From the CIF: _reflns_number_total 5626
Count of symmetry unique reflns 3631
Completeness (_total/calc) 154.94%
TEST3: Check Friedels for noncentro structure
Estimate of Friedel pairs measured 1995
Fraction of Friedel pairs measured 0.549

Are heavy atom types Z>Si present yes
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large.
0.12
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large.
5.52
PLAT301_ALERT_3_G Note: Main Residue Disorder .....
14.00 Perc.
PLAT860_ALERT_3_G Note: Number of Least-Squares Restraints .....
282
PLAT199_ALERT_1_G Check the Reported _cell_measurement_temperature
293 K
PLAT200_ALERT_1_G Check the Reported _diffrn_ambient_temperature
293 K
PLAT811_ALERT_5_G No ADDSYM Analysis: Too Many Excluded
Atoms .... !

---

1 ALERT level A = In general: serious problem
0 ALERT level B = Potentially serious problem
12 ALERT level C = Check and explain
8 ALERT level G = General alerts; check

4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
10 ALERT type 2 Indicator that the structure model may be wrong or deficient
3 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

# checkCIF/PLATON report (basic structural check)

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report

## Datablock: SCUTC-19

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Bond precision: C-C = 0.0127 A Wavelength=0.71073
Cell: a=10.925(2) b=10.946(2) c=13.972(3)
alpha=95.20(3) beta=99.23(3) gamma=99.89(3)
Temperature: 293 K
Calculated Reported

| | | |
|---|---|---|
| Volume | 1612.7(6) | 1612.7(5) |
| Space group | P -1 | P-1 |
| Hall group | -P 1 | -P 1 |
| Moiety formula | C28 H20 N2 O8 Zn2 | C28 H20 N2 O8 Zn2 |
| Sum formula | C28 H20 N2 O8 Zn2 | C28 H20 N2 O8 Zn2 |
| Mr | 643.24 | 643.20 |
| Dx,g cm-3 | 1.325 | 1.325 |
| Z | 2 | 2 |
| Mu (mm-1) | 1.532 | 1.532 |
| F000 | 652.0 | 652.0 |
| F000' | 653.41 | |
| h,k,lmax | 13,13,16 | 13,13,16 |
| Nref | 5805 | 5736 |
| Tmin,Tmax | 0.662,0.759 | 0.765,0.869 |
| Tmin' | 0.625 | |

Correction method= MULTI-SCAN

Data completeness= 0.988 Theta(max)= 25.200

R(reflections)= 0.0759( 2877) wR2(reflections)= 0.2137( 5736)

S = 0.941 Npar= 363

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The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

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## Alert level A

PLAT602_ALERT_2_A VERY LARGE Solvent Accessible VOID(S) in Structure !

---

## Alert level C

PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for C20
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for C23
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for C24
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for N2
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C22
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds (x 1000) Ang .. 13

PLAT369_ALERT_2_C Long C(sp2)-C(sp2) Bond C1 - C2 ...
1.53 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference O1 -- C1 ..
0.17 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference C3 -- C4 ..
0.17 Ang.

---

Alert level G
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large.
0.10
PLAT154_ALERT_1_G The su's on the Cell Angles are Equal (x 10000)
3000 Deg.
PLAT199_ALERT_1_G Check the Reported _cell_measurement_temperature
293 K
PLAT200_ALERT_1_G Check the Reported _diffrn_ambient_temperature
293 K
PLAT764_ALERT_4_G Overcomplete CIF Bond List Detected (Rep/Expd) .
1.12 Ratio
PLAT794_ALERT_5_G Note: Tentative Bond Valency for Zn1 .....
2.01
PLAT794_ALERT_5_G Note: Tentative Bond Valency for Zn2 .....
2.06

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8 ALERT type 2 Indicator that the structure model may be wrong or deficient
1 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
2 ALERT type 5 Informative message, check