SUPPLEMENTARY INFORMATION FILE ACCOMPANYING:

Interlinker hydrogen bonds govern CO₂ adsorption in a series of flexible 2D diaclyhydrazone/isophthalate-based MOFs: influence of metal center, linker substituent and activation temperature

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Figure S16 TGA curves for materials 1-4 conditioned in MeOH at room temperature for 1-2 days.
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Figure S20 The CO$_2$ (195 K) isotherm for material 1 soaked in MeOH and activated at 200 °C.
**Figure S21** CO$_2$ (195 K) and N$_2$ (77 K) isotherms for material 3 soaked in MeOH and activated at different temperatures.
Figure S22 PXRD patterns for 2 after various manipulations indicated on graphs. Activation was made in low pressure (~ $10^{-3}$ Pa).
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Table S1 Crystallographic data: comparison of bond lengths in coordination spheres for 1-4.

| Bond         | Bond lengths [Å] | 1     | 2     | 3b    | 4     |
|--------------|------------------|-------|-------|-------|-------|
| M01 – N01    |                  | 2.1580(16) | 2.1607(19) | 2.313(3) | 2.338(6) |
| M01 – N26    |                  | 2.1858(16) | 2.191(2)  | 2.315(3) | 2.344(6) |
| M01 – O37    |                  | 2.0129(15) | 2.0252(16) | 2.283(3) | 2.266(5) |
| M01 – O39    |                  | -      | -      | 2.393(2) | 2.443(4) |
| M01 – O40    |                  | 2.0030(13) | 1.9973(15) | 2.259(2) | 2.236(4) |
| M01 – O41    |                  | 2.0391(14) | 2.0404(16) | 2.387(2) | 2.338(4) |

M = Zn$^{2+}$ for 1-2 and M = Cd$^{2+}$ for 3b-4.

Table S2 Intra and interlayer π···π interactions for 1-4.

| Ring | Displacement [Å] | 1     | 2     | 3b    | 4     |
|------|------------------|-------|-------|-------|-------|
|      |                  | 1     | 2     | 3b    | 4     |
| R1   | R2               | 1.027 | 0.768 | 0.806 | 0.779 |
| R2   | R1               | 2.119 | 1.689 | 1.214 | 1.028 |
| R1   | R3               | -     | -     | 3.294 | 2.461 |
| R3   | R1               | -     | -     | 3.778 | 1.687 |
| R2   | R3               | 1.670 | 2.211 | -     | -     |
| R3   | R2               | 1.345 | 1.892 | -     | -     |
| R3   | R3               | -     | 1.740 | 2.002 | 2.136 |
Table S3 Selected hydrogen bond geometry (Å, °).

| D—H···A          | D—H | H···A | D···A | D—H···A |
|------------------|-----|-------|-------|---------|
| **1 (CCDC 1877585)** |
| N(9)-H(9N)...O(39) | 0.81(2) | 2.09(2) | 2.877(2) | 164(2) |
| N(20)-H(20N)...O(47) | 0.87(3) | 1.95(3) | 2.801(3) | 169(2) |
| -x-1/2, y-1/2, -z+1/2 |
| **2 (CCDC 1878988)** |
| N(9)-H(9N)...O(39)#1 | 0.82(3) | 2.02(3) | 2.808(3) | 161(3) |
| N(20)-H(20N)...O(47)#2 | 0.87(3) | 1.98(3) | 2.841(3) | 173(3) |
| (CCDC 1878988) |
| N(42)-H(42A)...O(8)#3 | 0.86(3) | 2.19(3) | 2.999(3) | 159(3) |
| N(42)-H(42B)...O(22)#4 | 0.82(4) | 2.16(4) | 2.984(3) | 175(4) |
| #1 -x+1,-y+2,-z+1 #2 -x+1,-y+1,-z+1 #3 -x+2,-y+2,-z+1 #4 x,y+1,z |
| **2MeOH (CCDC 1975137)** |
| N(9)-H(9N)...O(39)#1 | 0.86 | 2.02 | 2.790(6) | 148.3 |
| N(20)-H(20N)...O(50)#2 | 0.86 | 2.05 | 2.878(7) | 161.8 |
| N(42)-H(42B)...O(22)#3 | 0.86 | 2.14 | 2.981(7) | 166.1 |
| N(42)-H(42A)...O(8)#4 | 0.86 | 2.20 | 3.040(6) | 163.6 |
| #1 -x+2,-y+1,-z #2 -x+1,-y+1,-z+1 #3 x+1,y,z #4 -x+2,-y+2,-z |
| **3 (CCDC 1993922)** |
| N(2)-H(2)...O(18) | 0.86 | 2.10 | 2.94(2) | 165 |
| N(5)-H(5)...O(6)#1 | 0.86 | 2.05 | 2.854(9) | 154 |
| N(8)-H(8)...O(17) | 0.86 | 2.09 | 2.930(19) | 165 |
| N(11)-H(11)...O(10)#2 | 0.86 | 2.10 | 2.864(9) | 148 |
| #1 2-x,-y,1-z #2 -x,1-y,1-z |
| **3b (CCDC 1898831)** |
| N(20)-H(20N)...O(39)#1 | 0.82(4) | 2.04(4) | 2.843(4) | 163(3) |
| N(9)-H(9N)...O(47)#2 | 0.89(4) | 2.06(4) | 2.933(5) | 170(4) |
| #1 -x+1,-y,z #2 -x,-y,z+1 |
| **4 (CCDC 1975135)** |
| N(9)-H(9N)...O(47)#1 | 0.897(6) | 2.096(6) | 2.932(8) | 154.5(4) |
| N(20)-H(20N)...O(39)#2 | 1.005(5) | 1.866(4) | 2.841(7) | 162.7(3) |
| N(42)-H(42B)...O(8)#3 | 1.011(6) | 1.972(5) | 2.964(8) | 166.7(4) |
| N(42)-H(42A)...O(22)#4 | 1.014(6) | 2.271(5) | 3.060(8) | 133.6(4) |
| #1 -x+1,-y,-z #2 -x+2,-y,-z #3 x+1,y,z #4 -x+2,-y+1,-z |
Table S4 Comparison of selected IR absorption bands recorded for the as-synthesized, water soaked, and activated (200 °C, 50 mbar) materials 1-4.

| compound | (OH)$_{\text{H}_2\text{O}}$ | (NH)$_{\text{dih}}$ | (CO)$_{\text{DMF}}$ | (CO)$_{\text{dih}}$ | (COO)$_{\text{Xiso}}$ (asym, sym) |
|----------|----------------|----------------|----------------|----------------|----------------------------------|
| 1 as H$_2$O | 3350-3550 | 3249 | 1674 | 1665 | 1558, 1390 |
| act | - | 3260 | 1682 | 1655 | 1557, 1390 |
| 2 as H$_2$O | 3300-3550 | 3244 | - | 1652 | 1558, 1371 |
| act | - | 3208 | - | 1674 | 1558, 1386 |
| 3 as H$_2$O | 3300-3550 | 3241 | - | 1669 | 1558, 1387 |
| act | - | 3255 | 1674 | 1661 | 1557, 1387 |
| 4 as H$_2$O | 3300-3550 | 3220 | 1688$_{\text{sh}}$ | 1667 | 1557, 1385 |
| act | - | 3219 | - | 1669 | 1557, 1379 |

Table S5 Synthesis of MOFs 1-4: initial amounts and yields.

| MOF | Mass of substrate [mg] | Mass of product [mg] | Yield [%] |
|-----|------------------------|----------------------|-----------|
| M(NO$_3$)$_2$ | H$_2$Xiso | tdih | M(NO$_3$)$_2$ | H$_2$Xiso | tdih | 1 | 2 | 3 | 4 |
| 1as | 39.2 | 55.9 | 71.9 | 64.0 |
| 2as | 27.2 | 80.5 | 74.9 |
| 3as | 24.9 | 89.3 | 70.8 |
| 4as | 27.2 | 91.4 | 74.4 |
**Experimental Procedures**

Diffraction intensity data for single crystals of compounds 1-4 (except 2MeOH and 3) were collected on a KappaCCD (Nonius) diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). A suitably sized single crystal of 2MeOH prepared in a borosilicate glass capillary (d = 0.3 mm) with small amount of the mother liquor was measured on the Rigaku XtaLAB Synergy-S diffractometer with mirror-monochromated Mo Kα. Cell refinement and data reduction were performed using firmware. Positions of all of non-hydrogen atoms were determined by direct methods using SIR-97. All non-hydrogen atoms were refined anisotropically using weighted full-matrix least-squares on F^2. Refinement and further calculations were carried out using SHELXL 2014/7. All hydrogen atoms joined to carbon atoms were positioned with an idealized geometries and refined using a riding model with U_{iso}(H) fixed at 1.5 U_{eq} of C for methyl groups and 1.2 U_{eq} of C for other groups. The hydrogen atoms of the water (O66) molecule in 2 are indeterminate, H atoms attached to the N atoms were found in the difference-Fourier map and refined with an isotropic thermal parameter. Additionally, the crystal structure data shows that one DMF solvent molecule is heavily disordered and was removed using the SQUEEZE procedure implemented in the PLATON package. In case of other two DMF solvent molecules atoms were refined using DFIX and DANG instructions. The SQUEEZE procedure was also applied for 1 due to the presence of disordered guest molecules. The figures were made using CCDC1877585 (1), CCDC1878988 (2), CCDC1975137 (2MeOH) CCDC1898831 (3b) and CCDC1975135 (4) cif files that contain the supplementary crystallographic data.

A suitably sized single crystal of 3 was prepared in a borosilicate glass capillary (d = 0.3 mm) with small amount of the mother liquor. The dataset was collected at BESSY MX BL14.3 beamline of Helmholtz-Zentrum Berlin für Materialien und Energie. Monochromatic X-ray radiation with a wavelength of λ = 0.89500 Å (E = 13.85 keV) was used in experiments. The dataset was collected at room temperature. The crystal symmetry and scan range were determined in each particular case using iMosflm program. The φ-scans with oscillation range of 1° were used for data collection. For each dataset, 180 images were collected to reach the maximal possible completeness. The dataset was processed in automatic regime using XDSAPP 2.0 software. The Crystal structures were solved by direct methods and refined by full matrix least-squares on F^2 using SHELX-2018/3 program package. All non-hydrogen atoms were refined in anisotropic approximation. Hydrogen atoms were refined in geometrically calculated positions using “riding model” with U_{iso}(H)=1.2U_{iso}(C). Lattice water molecules O13-O21 were determined from the difference Fourier map and refined freely with restrains anisotropic parameters. The occupancy factor for O13, O18, O19 and O20 were refined and fixed in the last refinement cycle. The positions corresponding hydrogen atoms were determined from the electron density peaks and further refined with constrained isotropic parameter of U_{iso}(H)=1.2U_{iso}(O) and restrained geometry. The SQUEEZE procedure was applied to remove the contribution on refinement of the further lattice solvent molecules, which could not be located unambiguously. CCDC1993922 contains the supplementary crystallographic data for 3 Experimental data on single crystal X-ray experiments are summarized in Table S5.

CIFs files can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif
| Compounds | 1 | 2 | 2MeOH | 3 | 3b | 4 |
|-----------|---|---|-------|---|---|---|
| **Empirical formula** | C₆H₅N₂O₂Zn | C₆H₅N₂O₂Zn | C₆H₅N₂O₂Zn | C₆H₅N₂O₂Cd₂ | C₆H₆Cd₃N₂O₆ | C₆H₆Cd₃N₂O₆ |
| **Formula mass** | 674.96 | 689.98 | 664.93 | 1392.64 | 795.09 | 737.01 |
| **Temperature (K)** | 130(2) | 128(2) | 100.00(10) | 296(2) | 130(2) | 128(2) K |
| **Wave length (Å)** | 0.7107 | 0.71073 | 1.54184 | 0.89500 | 0.7107 | 0.71073 Å |
| **Crystal system** | Triclinic | Triclinic | Triclinic | Triclinic | Triclinic | Triclinic |
| **Space group** | P̅̅̅̅̅̅̅̅_̅̅̅ | P̅̅̅̅̅̅̅̅_̅̅̅ | P̅̅̅̅̅̅̅̅_̅̅̅ | P̅̅̅̅̅̅̅̅_̅̅̅ | P̅̅̅̅̅̅̅̅_̅̅̅ | P̅̅̅̅̅̅̅̅_̅̅̅ |
| **a (Å)** | 10.0351(3) | 9.9638(4) | 9.7787(5) | 10.4038(2) | 10.0911(2) | 10.1383(6) |
| **b (Å)** | 10.3247(2) | 10.9634(4) | 9.9374(3) | 14.9986(3) | 10.4145(2) | 10.3748(6) |
| **c (Å)** | 15.7932(4) | 16.7723(6) | 17.8681(6) | 24.4595(8) | 17.7975(3) | 17.7897(13) |
| **α (°)** | 99.740(2) | 76.666(3) | 86.483(3) | 82.5660(10) | 81.709(2) | 82.663(5) |
| **β (°)** | 93.331(2) | 86.693(3) | 79.981(3) | 86.7560(10) | 79.727(2) | 79.554(5) |
| **γ (°)** | 98.236(2) | 70.336(4) | 68.774(4) | 74.1180(10) | 75.010(2) | 75.940(5) |
| **Volume (Å³)** | 1590.41(7) | 1674.00(12) | 1593.85(12) | 3639.32(13) | 1768.31(6) | 1778.2(2) |
| **Z** | 2 | 2 | 2 | 2 | 2 | 2 |
| **Density (calculated) (g/cm³)** | 1.409 | 1.369 | 1.385 | 1.271 | 1.493 | 1.377 |
| **Absorption coefficient (mm⁻¹)** | 0.830 | 0.791 | 1.576 | 1.189 | 0.680 | 0.668 |
| **F(000)** | 696 | 712 | 684 | 1408 | 812 | 748 |
| **Crystal size (mm)** | 0.200 x 0.150 x 0.150 | 0.300 x 0.200 x 0.150 | 0.020 x 0.100 x 0.100 | 0.150 x 0.130 x 0.100 | 0.200 x 0.200 x 0.100 | 0.300 x 0.230 x 0.140 |
| **Theta range for data collection (°)** | 3.015 to 28.551 | 3.272 to 28.649 | 2.511 to 76.672 | 3.003 to 33.081 | 3.023 to 28.670 | 2.555 to 28.591 |
| **Index ranges** | -12 ≤ h ≤ 13, -12 ≤ k ≤ 13, -20 ≤ l ≤ 21 | -12 ≤ h ≤ 13, -13 ≤ k ≤ 14, -21 ≤ l ≤ 22 | -12 ≤ h ≤ 12, -17 ≤ k ≤ 17, -26 ≤ l ≤ 25 | -13 ≤ h ≤ 13, -13 ≤ k ≤ 13, -22 ≤ l ≤ 22 | -13 ≤ h ≤ 13, -13 ≤ k ≤ 13, -22 ≤ l ≤ 22 | -13 ≤ h ≤ 13, -13 ≤ k ≤ 13, -22 ≤ l ≤ 22 |
| **Reflection measured** | 22107 | 11699 | 42325 | 20689 | 24094 | 12653 |
| **Reflections unique** | 7338 [R(int) = 0.0283] | 7483 [R(int) = 0.0241] | 6437 [R(int) = 0.0619] | 9892 [R(int) = 0.0823] | 8216 [R(int) = 0.0328] | 7927 [R(int) = 0.0422] |
| **Completeness theta = 25.241°** | 99.80% | 99.40% | 99.6% | 71.50% | 99.60% | 99.80% |
| **Data / restraints / parameters** | 7338 / 0 / 425 | 7483 / 0 / 442 | 6437 / 0 / 409 | 9892 / 18 / 834 | 8216 / 12 / 472 | 7927 / 5 / 394 |
| **Goodness-of-fit on F2** | 1.030 | 1.036 | 1.710 | 1.098 | 1.022 | 0.848 |
| **Final R indices** | R1 = 0.0362, wR2 = 0.0813 | R1 = 0.0395, wR2 = 0.0982 | R1 = 0.1348, wR2 = 0.3574 | R1 = 0.0826, wR2 = 0.2452 | R1 = 0.0446, wR2 = 0.1146 | R1 = 0.0802, wR2 = 0.2108 |
| **R indices (all data)** | R1 = 0.0457, wR2 = 0.0868 | R1 = 0.0512, wR2 = 0.1061 | R1 = 0.1409, wR2 = 0.3643 | R1 = 0.0575, wR2 = 0.2574 | R1 = 0.0575, wR2 = 0.1231 | R1 = 0.1126, wR2 = 0.2422 |
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