

Supporting Information

Mechanical bonding of rigid MORFs using a tetratopic rotaxane

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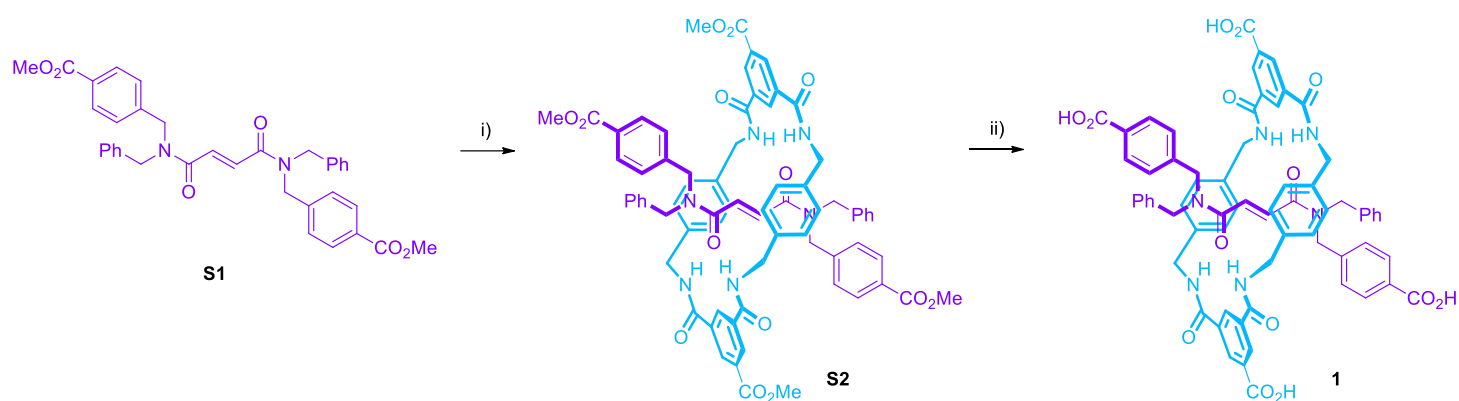
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1. General experimental section

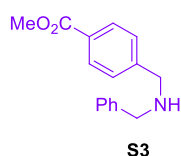
Unless stated otherwise, all reagents were purchased from Aldrich Chemicals and used without further purification. HPLC grade solvents (Scharlab) were nitrogen saturated and were dried and deoxygenated using an Innovative Technology Inc. Pure-Solv 400 Solvent Purification System (Pure Solvent Solutions, Herten, Netherlands). Column chromatography was carried out using silica gel (60 Å, 70-200 µm, SDS) as stationary phase, and TLC was performed on precoated silica gel on aluminum cards (0.25 mm thick, with fluorescent indicator 254 nm, Fluka) and observed under UV light. All melting points were determined on a Kofler hot-plate melting point apparatus and are uncorrected. ¹H- and ¹³C-NMR spectra were recorded at 298 K on a Bruker Avance 300 and 400 MHz instruments (Bruker, Massachusetts, USA). ¹H-NMR chemical shifts are reported relative to Me₄Si and were referenced via residual proton resonances of the corresponding deuterated solvent, whereas ¹³C NMR spectra are reported relative to Me₄Si using the carbon signals of the deuterated solvent. Abbreviations of coupling patterns are as follows: br, broad; s, singlet; d, doublet; t, triplet; q, quadruplet; m, multiplet. Coupling constants (*J*) are expressed in Hz. High-resolution mass spectra (HRMS) were obtained using a time-of-flight (TOF) instrument equipped with electrospray ionization (ESI). Scanning Electron Microscopy (SEM) images were obtained with a JEOL JSM-6100 Scanning Microscope (200 kV) (JEOL, Tokyo, Japan). SEM samples were prepared by deposition of corresponding MOFs on a glass sample holder and subsequent coated with gold. Optical microscope images were obtained with a 48-megapixel Sony IMX586 quad camera attached to a stereomicroscope OPTIKA SLX-1 (OPTIKA Italy, Ponterarica, Italy). The infrared spectra of the different compounds were measured using a JASCO ATR FTIR-4700 (JASCO Analytica Spain, Madrid, Spain). Microanalyses were performed on a Leco CHNS-932 Elemental Analyser (Leco, Michigan, USA). The thermogravimetric analyses were performed on a SDT 2960 simultaneous DTA-TGA analyser (TA Instruments, New Castle, USA). X-ray Powder Diffraction Analysis (XRPD) pattern of grinded crystals was collected in a Bruker D8 ADVANCE Powder X-ray diffractometer (Bruker, Massachusetts, USA). Single crystal X-ray structures were collected in a Bruker D8QUEST diffractometer (Bruker, Massachusetts, USA). CO₂ adsorption experiments were carried out using a surface area and pore size analyzer Autosorb 6iSA QUANTACHROME (Quantachrome corporation, Florida, USA).

2. Synthesis of tetratopic ligand **1**



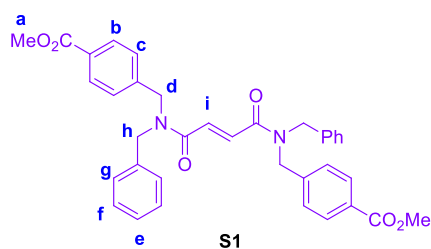
Scheme S1. Synthesis of interlocked tetraacid **1** and the corresponding intermediate. Reaction conditions: i) *p*-xylylenediamine, 5-(methoxycarbonyl)isophthaloyl chloride, Et₃N, CHCl₃, 25 °C, 4 h; ii) LiOH·H₂O, H₂O/DMF/THF (1:1.5:3), 70 °C, 16 h, then neutralization with HCl 6M.

Amine **S3**



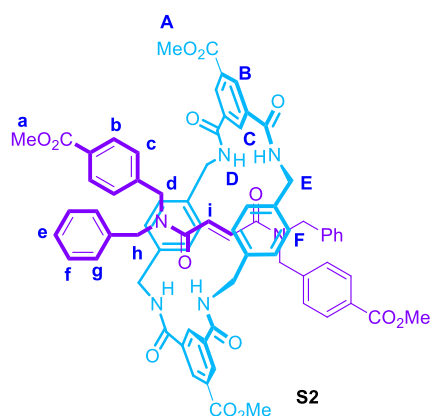
The synthesis and characterization of **S3** are described in the following reference: D. Chianelli, P. V. Rucker, J. Roland, D. C. Tully, J. Nelson, X. Liu, B. Bursulaya, E. D. Hernandez, J. Wu, M. Prashad, T. Schalma, Y. Liu, A. Chu, J. Schmeits, D. J. Huang, R. Hill, D. Bao, J. Zoll, Y. Kim, T. Groessl, P. McNamara, B. Liu, W. Richmond, I. Sancho-Martinez, A. Phimister, H. M. Seidel, M. K. Badman, S. B. Joseph, B. Laffitte, V. Molteni, *J. Med. Chem.* **2020**, *63*, 3868-3880.

Thread S1



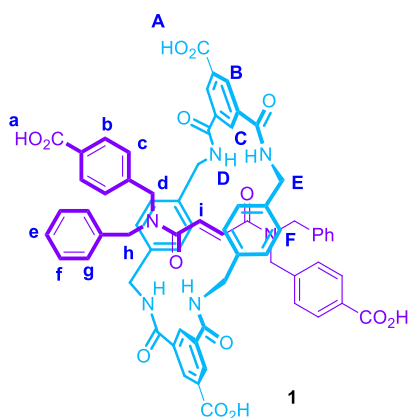
A solution of fumaryl chloride (0.7 mL, 6.54 mmol) in chloroform (50 mL) was added dropwise to a solution of amine **S3** (3.67 g, 14.38 mmol) and Et₃N (3.2 mL, 22.88 mmol) in chloroform (100 mL) under N₂ atmosphere at 0 °C. The resulting mixture was stirred for 24 hours at room temperature and then, washed with HCl 1 M (2 x 150 mL), sat. NaHCO₃ (2 x 150 mL) and brine (1 x 100 mL). The organic phase was dried over anhydrous MgSO₄ and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel using a CHCl₃/acetone (95/5) mixture as eluent, to give the title product as a white solid (**S1**, 3.15 g, 82 %); m.p. 129-131 °C; ¹H NMR (400 MHz, CDCl₃, mixture of rotamers) δ (ppm) = 8.04-7.97 (m, 4H, H_b), 7.61-7.13 (m, 16H, H_{c+g+e+i}), 4.68-4.58 (m, 8H, H_{d+h}), 3.94 (s, 3H, H_a), 3.91 (s, 3H, H_a); ¹³C NMR (101 MHz, CDCl₃, mixture of rotamers) δ (ppm) = 166.9 (CO), 166.7 (CO), 165.9 (CO), 165.8 (CO), 165.8 (CO), 142.0 (C), 141.3 (C), 136.4 (C), 135.7 (C), 132.5 (CH), 132.4 (CH), 130.4 (CH), 130.1 (CH), 130.0 (CH), 129.6 (CH), 129.2 (CH), 128.9 (CH), 128.5 (CH), 128.2 (CH), 127.9 (CH), 126.8 (CH), 126.8 (CH), 126.7 (CH), 52.4 (CH₃), 52.3 (CH₃), 50.6 (CH₂), 50.1 (CH₂), 49.0 (CH₂), 48.6(CH₂). HRMS (ESI) calculated for C₃₆H₃₅N₂O₆ [M + H]⁺ 591.2495, found 591.2490 (-0.84 ppm).

Rotaxane S2



Thread **S1** (1.5 g, 2.54 mmol) and Et₃N (5.5 mL, 39.89 mmol) in anhydrous CHCl₃ (400 mL) were stirred vigorously whilst solutions of *p*-xylylenediamine (2.42 g, 17.68 mmol) in anhydrous CHCl₃ (20 mL) and 5-(methoxycarbonyl)isophthaloyl chloride (4.62 g, 17.68 mmol) in anhydrous CHCl₃ (20 mL) were simultaneously added over a period of 4 h using motor-driven syringe pumps. After the addition, the resulting suspension was filtered through a Celite[®] pad and the filtrate was washed with water (2 x 50 mL), a saturated solution of NaHCO₃ (2 x 50 mL), a solution of HCl (1M) and brine (2 x 50 mL). The organic phase was then dried over MgSO₄, filtered and the solvent removed under reduced pressure. The resulting residue was subjected to column chromatography (silica gel) using a CH₂Cl₂:MeOH mixture (98:2) to yield the target [2]rotaxane (**S2**, 0.67 g, 21%). m.p. > 200 °C; ¹H NMR (400 MHz, CDCl₃, mixture of rotamers) δ (ppm) = 8.81-8.77 (m, 4H, H_B), 8.53-8.44 (m, 2H, H_C), 8.01 (t, *J* = 7.6 Hz, 2H, H_D), 7.63-6.69 (m, 24H, H_{D+F+b+c+e+f+g}), 6.11-5.90 (m, 2H, H_i), 5.16 (bs, 4H, H_E), 4.67-4.24 (m, 8H, H_{d+h}), 4.02-3.91 (m, 12H, H_{A+a}), 3.46 (bs, 4H, H_{E'}); ¹³C NMR (101 MHz, CDCl₃, mixture of rotamers) δ (ppm) = 166.3 (CO), 166.1 (CO), 165.9 (CO), 165.8 (CO), 164.4 (CO), 164.3 (CO), 164.2 (CO), 140.4 (C), 140.3 (C), 138.9 (C), 138.7 (C), 138.3 (C), 138.2 (C), 138.1 (C), 135.3 (CH), 135.2 (CH), 133.9 (CH), 133.8 (CH), 133.2 (CH), 132.3 (CH), 132.0 (CH), 130.8 (CH), 130.5 (CH), 130.3 (CH), 130.2 (CH), 130.1 (CH), 130.0 (CH), 129.8 (CH), 129.6 (CH), 129.3 (CH), 129.3 (CH), 129.1 (CH), 129.0 (CH), 128.8 (CH), 125.7 (CH), 125.6 (CH), 125.4 (CH), 52.8 (CH₃), 52.7 (CH₃), 52.5 (CH₃), 51.9 (CH₂), 51.4 (CH₂), 51.2 (CH₂), 51.1 (CH₂), 50.9 (CH₂), 43.4 (CH₂), 43.3 (CH₂). HRMS (ESI) calculated for C₇₂H₆₇N₆O₁₄ [M + H]⁺ 1239.4715, found 1239.4735 (+1.62 ppm).

Rotaxane 1



A solution of LiOH·H₂O (0.068 g, 1.6 mmol) in H₂O (2 mL) was added to a solution of rotaxane **S2** (0.100 g, 0.08 mmol) in DMF (3 mL) and THF (6 mL), and heated at 70 °C for 16 h. After that time, THF was removed under reduced pressure and HCl was added until pH 1 was reached. The solvent was removed by filtration and the resulting solid was extensively washed with water, chloroform and diethyl ether. The product was obtained as a white solid (**1**, 0.090 g, 95%). m.p. > 200 °C; ¹H NMR (400 MHz, DMSO-*d*₆, mixture of rotamers) δ (ppm) = 13.16 (bs, 4H, H_{A+a}), 8.67-8.18 (m, 12H, H_{B+C+b+e}), 7.84-6.91 (m, 24H, H_{D+F+c+f+g}), 6.10-5.94 (m, 2H, H_i), 4.48-4.06 (m, 16H, H_{E+d+h}); ¹³C NMR (101 MHz, DMSO-*d*₆, mixture of rotamers) δ (ppm) = 167.0 (CO), 166.6 (CO), 166.3 (CO), 165.5 (CO), 165.4 (CO), 164.1 (CO), 164.0 (CO), 163.9 (CO), 141.3 (C), 140.0 (C), 139.9 (C), 137.6 (C), 136.1 (C), 135.0 (CH), 134.9 (CH), 134.2 (CH), 134.1 (CH), 134.0 (CH), 132.0 (CH), 131.7 (CH), 131.1 (CH), 129.9 (CH), 129.8 (CH), 129.7 (CH), 129.3 (CH), 129.2 (CH), 129.1 (CH), 129.0 (CH), 128.9 (CH), 128.8 (CH), 128.7 (CH), 128.4 (CH), 128.3 (CH), 128.2 (CH), 127.7 (CH), 127.5 (CH), 127.4 (CH), 126.3 (CH), 126.2 (CH), 51.1 (CH₂), 50.7 (CH₂), 49.8 (CH₂), 49.7 (CH₂), 49.5 (CH₂), 42.9 (CH₂). HRMS (ESI) calculated for C₆₈H₅₉N₆O₁₄ [M + H]⁺ 1183.4084, found 1183.4064 (-1.69 ppm).

3. Synthesis of UMUMOFs-7 and -8

The tetratopic ligand **1** was combined with the metallic salt in a mixture of solvents DMF:EtOH:H₂O (3:3:2) with one drop of HNO₃. This solution was filtered by using EMD Millipore Millex™ Sterile Syringe Filters 0.45 μm pore size and distributed in borosilicate glass vials with GL18 screw cap. The solution was then heated using the heating ramp shown in the graph below (Figure S1): heating from 20 °C to 80 °C for 15 minutes, then this temperature was maintained for 48 hours and, finally, a slow cooling process for 24 hours. The obtained crystals were filtered and exhaustively washed with DMF and CHCl₃, and then dried under vacuum.

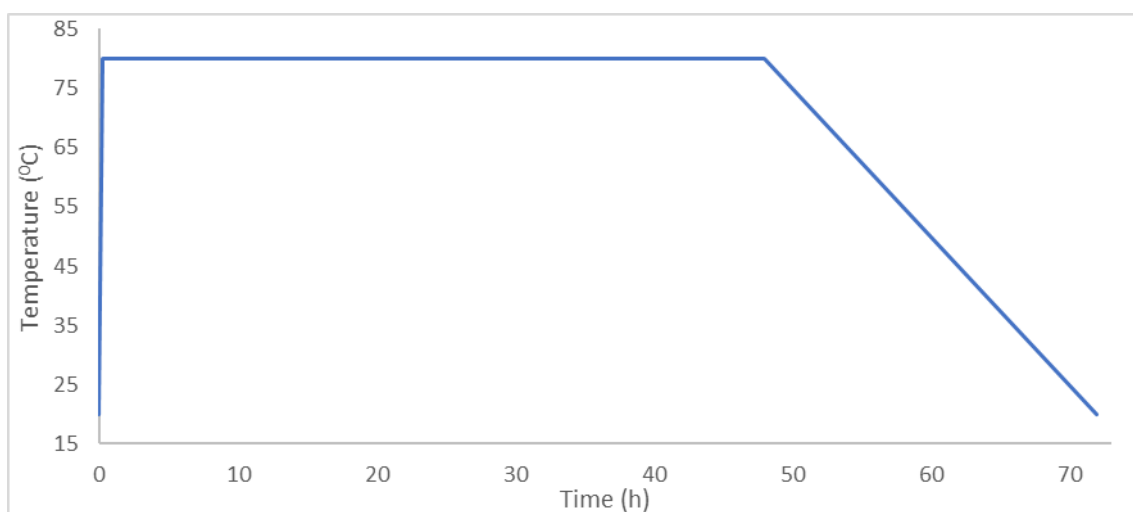


Figure S1. Heating ramp program.

UMUMOF -7

A solution of Cu(NO₃)₂·H₂O (10.5 mg, 0.056 mmol) in EtOH (1.7 mL), water (1.1 mL) and a drop of HNO₃ was added to a solution of rotaxane **1** (16.5 mg, 0.014 mmol) in DMF (1.7 mL). The general procedure was followed, obtaining blue crystals (**UMUMOF-7**, 24 mg, 64%). Elemental Analysis (C₆₈H₅₈Cu₂N₆O₁₆·1.95 C₃H₇NO·1.2 CHCl₃, %) found (calculated): C 54.57 (54.58), H 4.59 (4.60), N 6.73 (6.74). FTIR (ATR, cm⁻¹) ν = 3334 (w, NH), 1651 (vs, C-OCu), 1594 (vs, C-OCu), 1358 (s, ArC-O), 1331 (s, ArC-O).

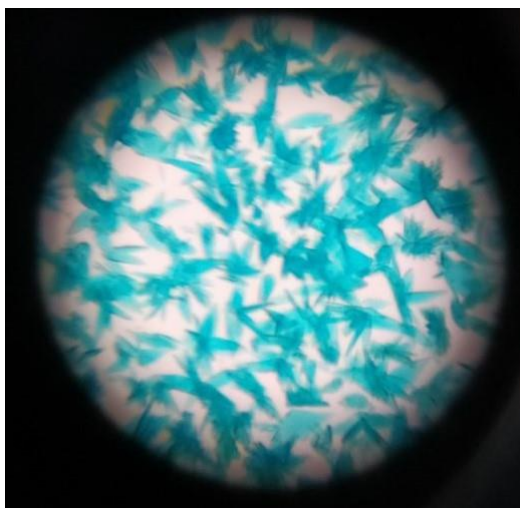


Figure S2. Photograph of **UMUMOF-7** crystals observed using an optical microscope.

UMUMOF-8

A solution of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (20.8 mg, 0.072 mmol) in EtOH (1.1 mL), water (0.7 mL) and a drop of HNO_3 was added to a solution of rotaxane **1** (10.57 mg, 0.009 mmol) in DMF (1.1 mL). The general procedure was followed, obtaining reddish crystals (**UMUMOF-8**, 15 mg, 60%). Elemental Analysis ($\text{C}_{68}\text{H}_{60}\text{Co}_2\text{N}_6\text{O}_{19} \cdot 3.05 \text{C}_3\text{H}_7\text{NO} \cdot 2.5 \text{C}_2\text{H}_6\text{O} \cdot 2.04 \text{CHCl}_3$, %) found (calculated): C 54.47 (56.46), H 5.40 (5.39), N 6.83 (6.83). FTIR (ATR, cm^{-1}) $\nu = 3339$ (w, NH), 1650 (vs, C-OCO), 1597 (vs, C-OCO), 1351 (s, ArC-O), 1343 (s, ArC-O).

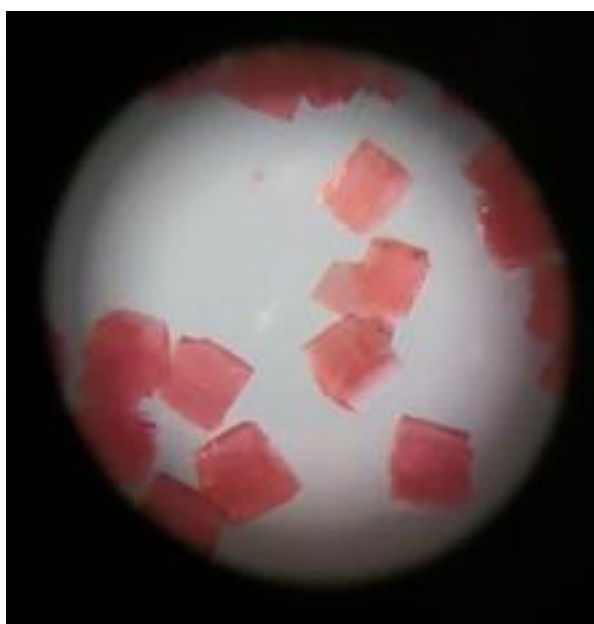


Figure S3. Photograph of **UMUMOF-8** crystals observed using an optical microscope.

4. Scanning Electron Microscopy

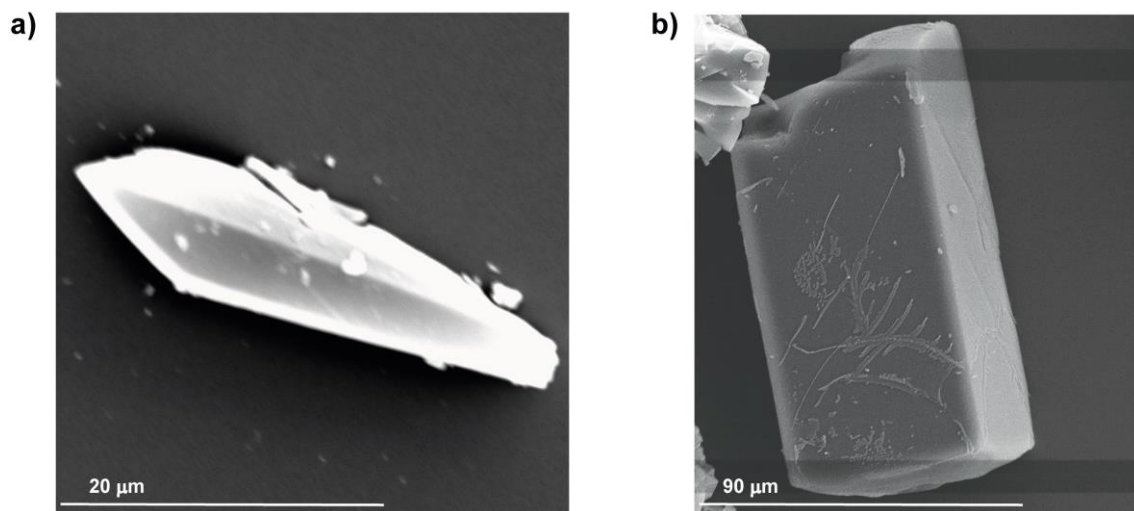


Figure S4. SEM micrographs of a: (a) UMUMOF-7 crystal; (b) UMUMOF-8 crystal.

5. Thermogravimetric Analysis

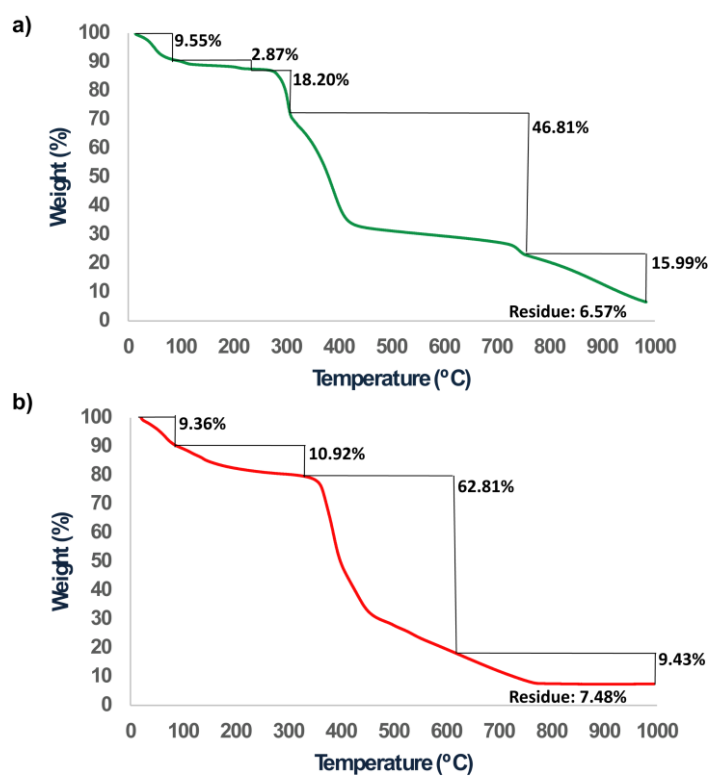


Figure S5. Thermogravimetric analyses (5 °C/minute, N₂ stream) of UMUMOF-7 (a) and UMUMOF-8 (b).

6. Adsorption experiments

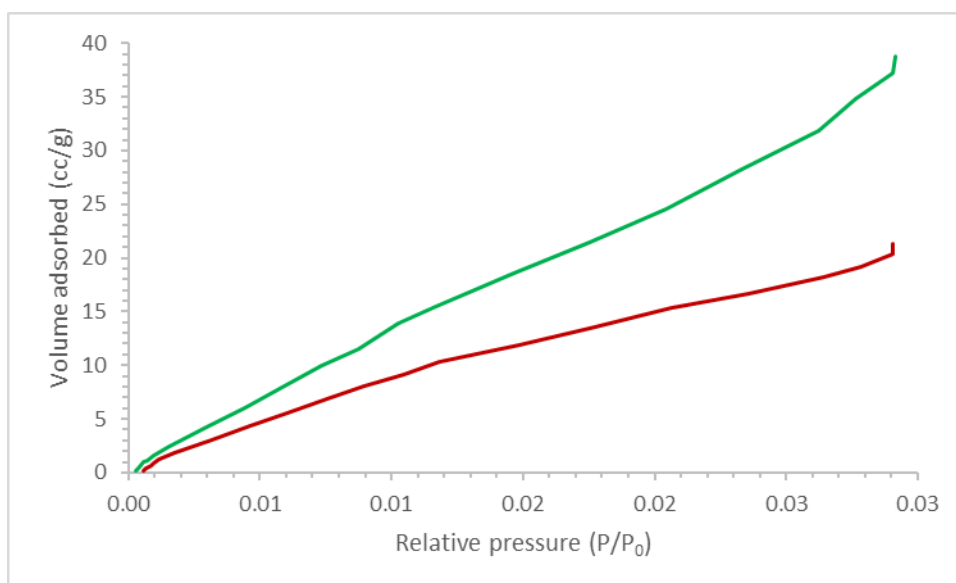


Figure S6. CO₂ adsorption isotherms ($P_{0,override} = 26142$ Torr, outgas time = 4 h, $T_{outgas} = 523$ K, $T_{bath} = 273$ K) of **UMUMOF-7** (green) and **UMUMOF-8** (red).

Table S1. CO₂ DR adsorption data of **UMUMOFs-7 and -8**.

Measurement	UMUMOF-7	UMUMOF-8
Pore surface area (m ² /g)	295.959	333.905
Pore volume (cc/g)	0.101	0.114
Correlation coefficient	0.9995	0.9997

7. X-ray Powder Diffraction Analysis

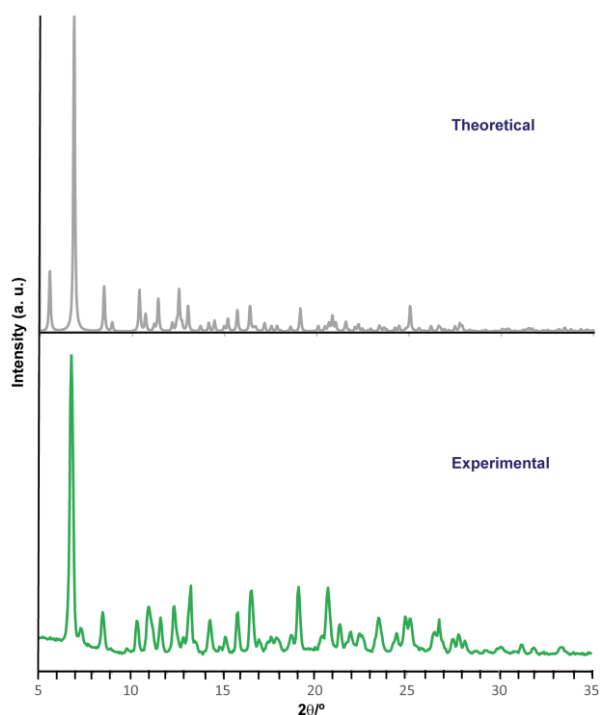


Figure S7. Theoretical (grey) and experimental (green) XRPD pattern of **UMUMOF-7** (Cu K_{α} = 1.5406 Å, electrical current = 40 mA, electric power = 45 kV, step size = 0.015°).

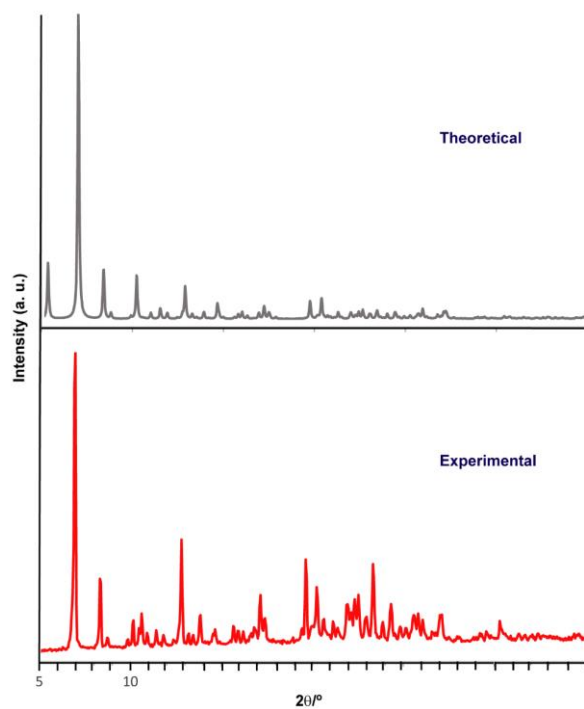


Figure S8. Theoretical (grey) and experimental (red) XRPD pattern of **UMUMOF-8** (Cu K_{α} = 1.5406 Å, electrical current = 40 mA, electric power = 45 kV, step size = 0.015°).

8. Infrared Spectroscopy

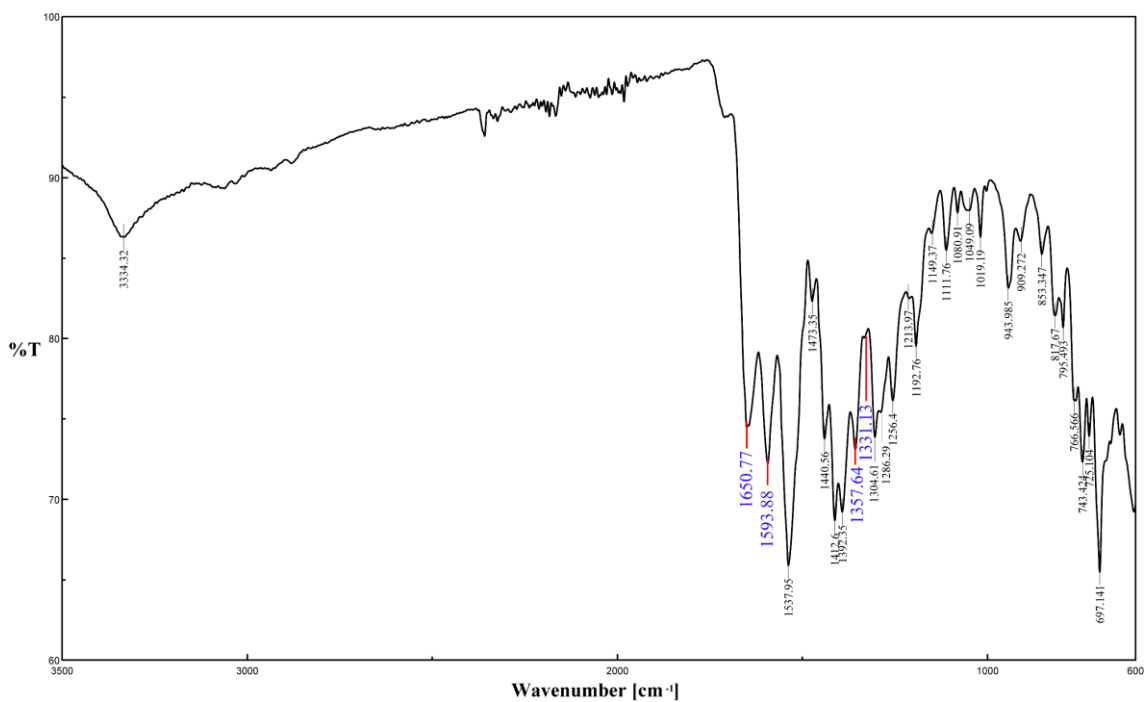


Figure S9. Infrared spectrum (ATR) of **UMUMOF-7**, highlighting in blue the asymmetric and symmetric stretching vibration of carboxylate-copper coordination bonds.

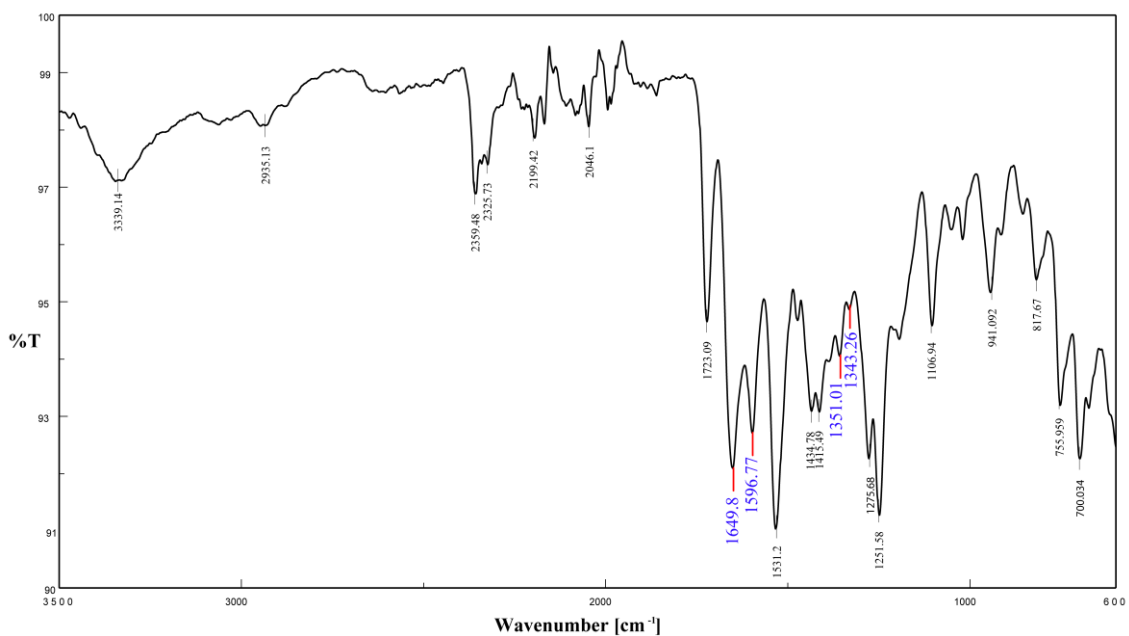


Figure S10. Infrared spectrum (ATR) of **UMUMOF-8**, highlighting in blue the asymmetric and symmetric stretching vibration of carboxylate-cobalt coordination bonds.

9. Crystal data and structure refinement for UMUMOFs-7 and -8

UMUMOF-7

Single crystals of $C_{68}H_{58}Cu_2N_6O_{16}$ [As_III_140_0msp_a] were obtained by following the above described procedure. Intensities were registered at low temperature (100.0 K) on a Bruker D8 QUEST system equipped with a multilayer monochromator and a Mo K/ α Incoatec microfocus sealed tube ($\lambda = 0.71073 \text{ \AA}$). Absorption corrections were based on multi-scans (program SADABS). Using Olex2,¹ the structure was solved with the SHELXT² structure solution program using Intrinsic Phasing and refined with the SHELXL³ refinement package using Least Squares minimization. Hydrogen atoms were included using a riding model. A solvent mask was calculated and 520 electrons were found in a volume of 2197 \AA^3 in 1 void per unit cell. This is consistent with the presence of 6 molecules of H_2O per Asymmetric Unit, which account for 480 electrons per unit cell. The structure was deposited with CSD (deposition number CCDC 2340441).

Table S2. Crystal data and structure refinement for **UMUMOF-7** (CCDC 2340441).

Bond precision:	C-C = 0.0052 \AA	Wavelength = 0.71073 \AA	
Cell:	a = 32.307(2) \AA	b = 14.1238(11) \AA	c = 16.6531(12) \AA
	$\alpha = 90^\circ$	$\beta = 97.426(3)^\circ$	$\gamma = 90^\circ$
Temperature:	100 K		
	Calculated	Reported	
Volume	7535.0(9) \AA^3	7535.1(10) \AA^3	
Space group	<i>C2/c</i>	<i>C12/c1</i>	
Hall group	- <i>C2yc</i>	- <i>C2yc</i>	
Moiety formula	$C_{34}H_{29}CuN_3O_8$ [+solvent]	$C_{68}H_{58}Cu_2N_6O_{16}$	
Sum formula	$C_{34}H_{29}CuN_3O_8$ [+solvent]	$C_{68}H_{58}Cu_2N_6O_{16}$	
Mr	671.15 g/mol	1342.28 g/mol	
Dx, g cm⁻³	1.183	1.183	
Z	8	4	
μ (mm⁻¹)	0.627	0.627	
F000	2776.0	2776.0	
F000'	2779.80		
h,k,l_{max}	46,20,23	46,20,23	
N_{ref}	11559	11539	
T_{min}, T_{max}	0.956 K, 0.969 K	0.694 K, 0.746 K	

T_{min}	0.888 K
Correction method	= # Reported T Limits: T _{min} = 0.694 T _{max} = 0.746
AbsCorr =	
MULTI-SCAN	
Data	Θ _{max} = 30.555°
completeness =	
0.998	

UMUMOF-8

Single crystals of C₆₈H₆₀Co₂N₆O₁₉ [AS_IV__0msp] were obtained by following the above described procedure. Intensities were registered at low temperature (100.0 K) on a Bruker D8 QUEST system equipped with a multilayer monochromator and a Mo K/α Incoatec microfocus sealed tube (λ = 0.71073 Å). Absorption corrections were based on multi-scans (program SADABS). Using Olex2,¹ the structure was solved with the SHELXT² structure solution program using Intrinsic Phasing and refined with the SHELXL³ refinement package using Least Squares minimization. Hydrogen atoms were included using a riding model. One of the phenyl groups at the thread, originated due to a translation movement, is disordered, and was modelled. A solvent mask was calculated and 499 electrons were found in a volume of 2465 Å³ in 1 void per unit cell. This is consistent with the presence of 2 molecules of H₂O and 1 molecule of DMF per Asymmetric Unit, which account for 480 electrons per unit cell. The structure was deposited with CSD (deposition number CCDC 2340442).

Table S3. Crystal data and structure refinement for **UMUMOF-8** (CCDC 2340442).

Bond precision:	C-C = 0.0055 Å	Wavelength = 0.71073 Å	
Cell:	a = 16.7950(2) Å	b = 33.970(11) Å	c = 13.8988(12) Å
	α = 90°	β = 90°	γ = 90°
Temperature:	100 K		
	Calculated	Reported	
Volume	7929.6(8) Å ³	7929.6(8) Å ³	
Space group	<i>Pnma</i>	<i>Pnma</i>	
Hall group	<i>-P2ac2n</i>	<i>-P2ac2n</i>	
Moiety formula	C ₆₈ H ₆₀ Co ₂ N ₆ O ₁₉ [+solvent]	C ₆₈ H ₆₀ Co ₂ N ₆ O ₁₉	
Sum formula	C ₆₈ H ₆₀ Co ₂ N ₆ O ₁₉ [+solvent]	C ₆₈ H ₆₀ Co ₂ N ₆ O ₁₉	

Mr	1383.08 g/mol	1383.08 g/mol
Dx,g cm⁻³	1.158	1.159
Z	8	4
μ (mm⁻¹)	0.483	0.483
F000	2864.0	2864.0
F000'	2868.17	
h,k,l_{max}	22,45,18	22,45,18
N_{ref}	10048	10028
T_{min},T_{max}	0.935 K,0.981 K	0.660 K,0.746 K
T_{min}'	0.935 K	
Correction method= # Reported T Limits: T_{min} = 0.660 T_{max} = 0.746		
AbsCorr = MULTI-SCAN		
Data completeness =		Θ_{max} = 28.314°
0.998		

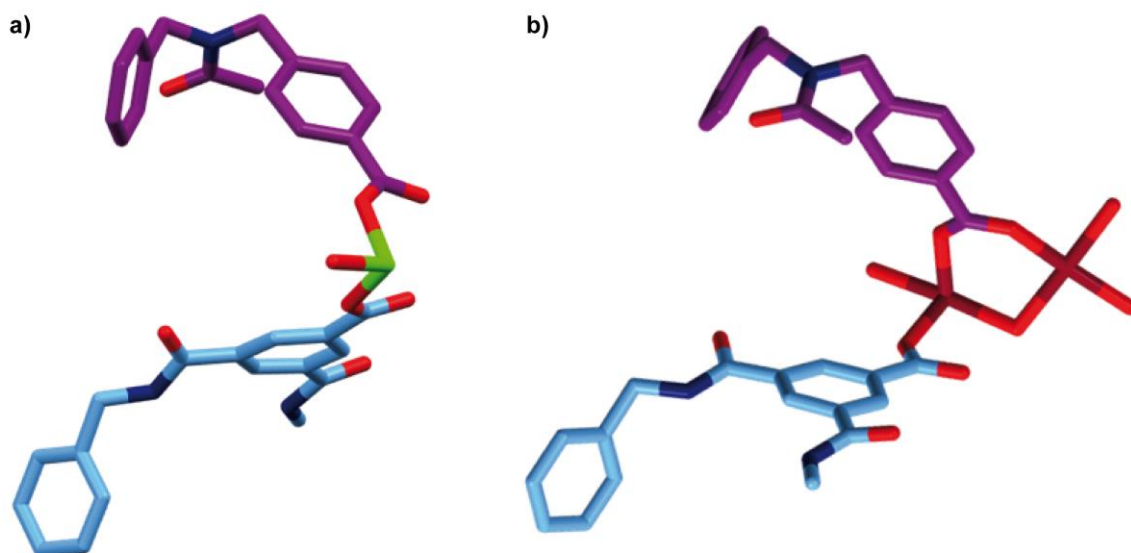


Figure S11. Stick representation of the asymmetric units of: (a) **UMUMOF-7** and (b) **UMUMOF-8**.

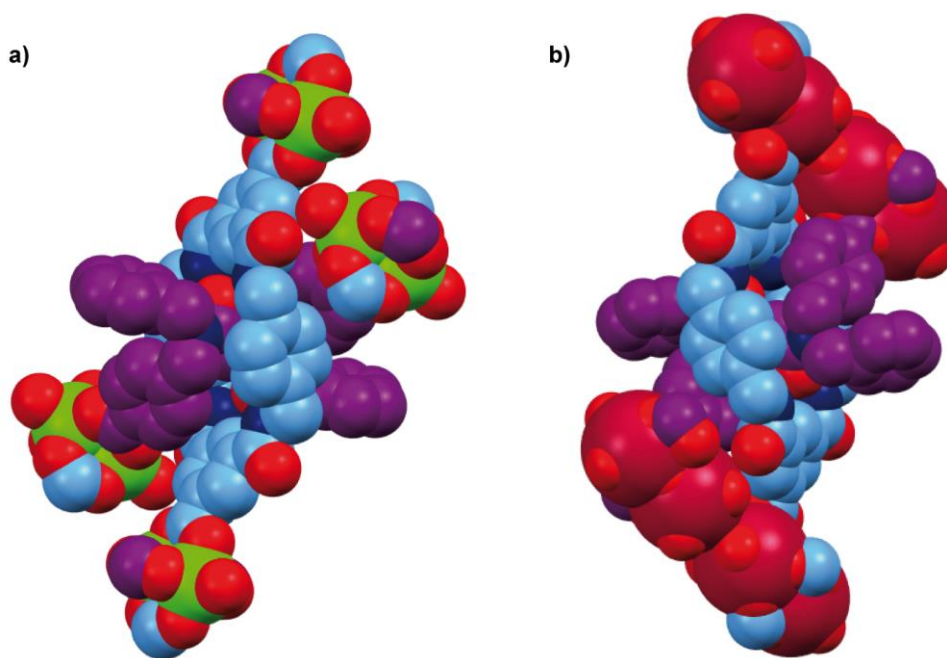


Figure S12. Spacefill representation of a single unit of tetratopic ligand **1** within the crystalline array of: (a) **UMUMOF-7** and (b) **UMUMOF-8**.

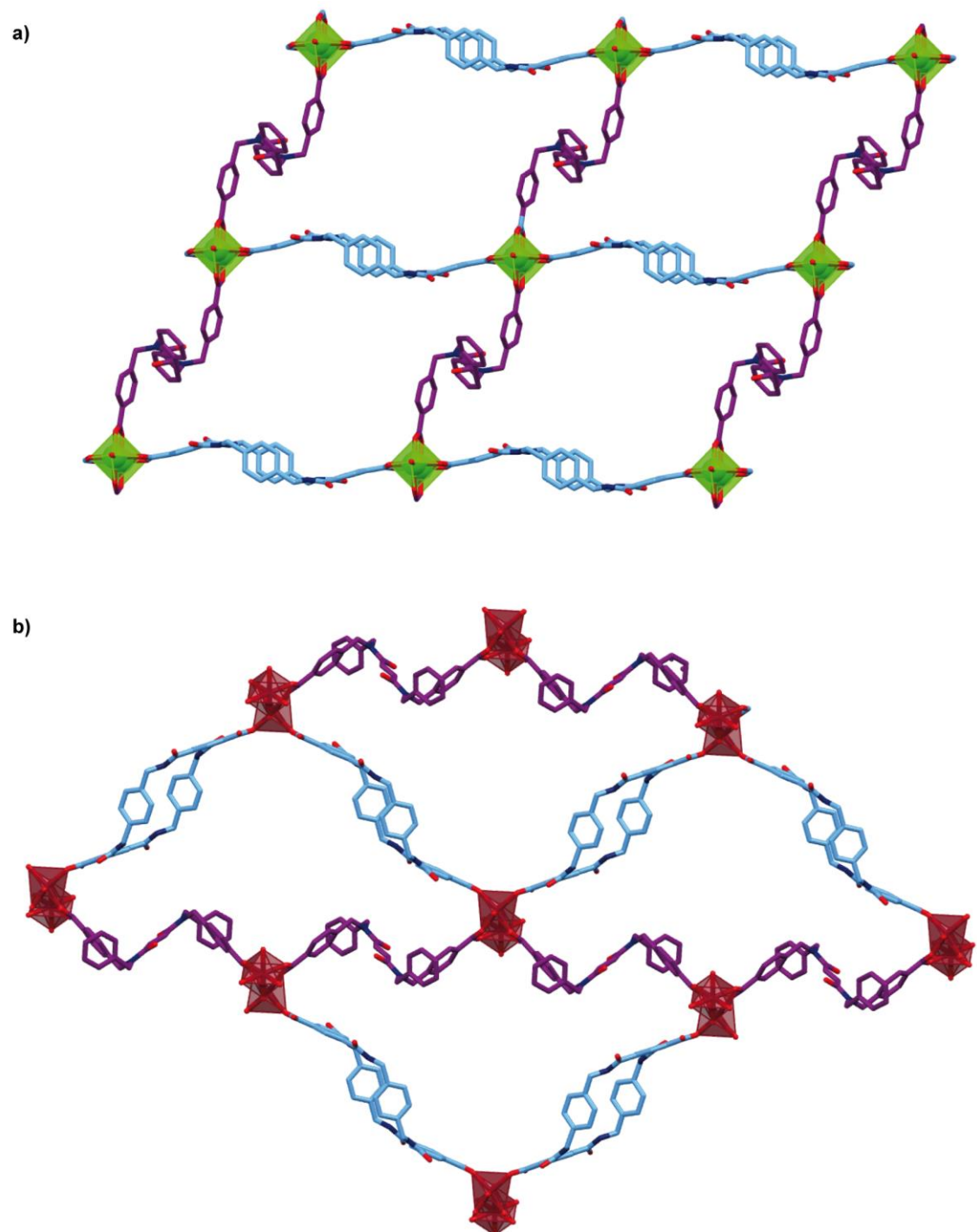


Figure S13. 2D-stick representation of four interconnected metallogrids of: (a) **UMUMOF-7** and (b) **UMUMOF-8**. Only one component of the interlocked ligand (thread or macrocycle) is shown for clarity.

10. X-ray diffraction analyses using different solvents

In order to study the influence of different solvents in the structure, a batch of **UMUMOF-8** crystals was dried under vacuum for 23 hours and a suitable crystal was selected and placed in the vacuum set of the X-ray Diffractometer, where an additional vacuum cycle was accomplished. The measurement was performed under nitrogen atmosphere to avoid atmospheric water uptake.

A second batch of crystals was washed three times with fresh ethanol and then dried under vacuum for 24 hours. After that time, the evacuated crystals were immersed in the corresponding solvent (ethanol or toluene) for 24 hours. Then, selected crystals were measured using the X-ray Diffractometer under nitrogen atmosphere. In the studies using bromobenzene as solvent, this solvent was dripped on selected crystals due to difficulties during the extraction of crystals directly immerse in bromobenzene.

Analogous experiments using **UMUMOF-7** were carried out, but the obtained crystallographic data did not allow the structural analysis of the crystals.

Table S4. Single crystal X-ray Diffraction analysis of dried **UMUMOF-8** using different solvents.

Solvent	Cell Parameters							Space group	Bravais lattice
	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	v (Å ³)		
-	16.854	34.039	13.827	90	90	90	7932.0	<i>Pnma</i>	Orthorhombic P
Ethanol	16.721	34.003	13.837	90	90	90	7867.3	<i>Pnma</i>	Orthorhombic P
Toluene	16.729	33.928	13.861	90	90	90	7867.2	<i>Pnma</i>	Orthorhombic P
Bromobenzene	16.821	34.219	13.905	90	90	90	8004.1	<i>Pnma</i>	Orthorhombic P

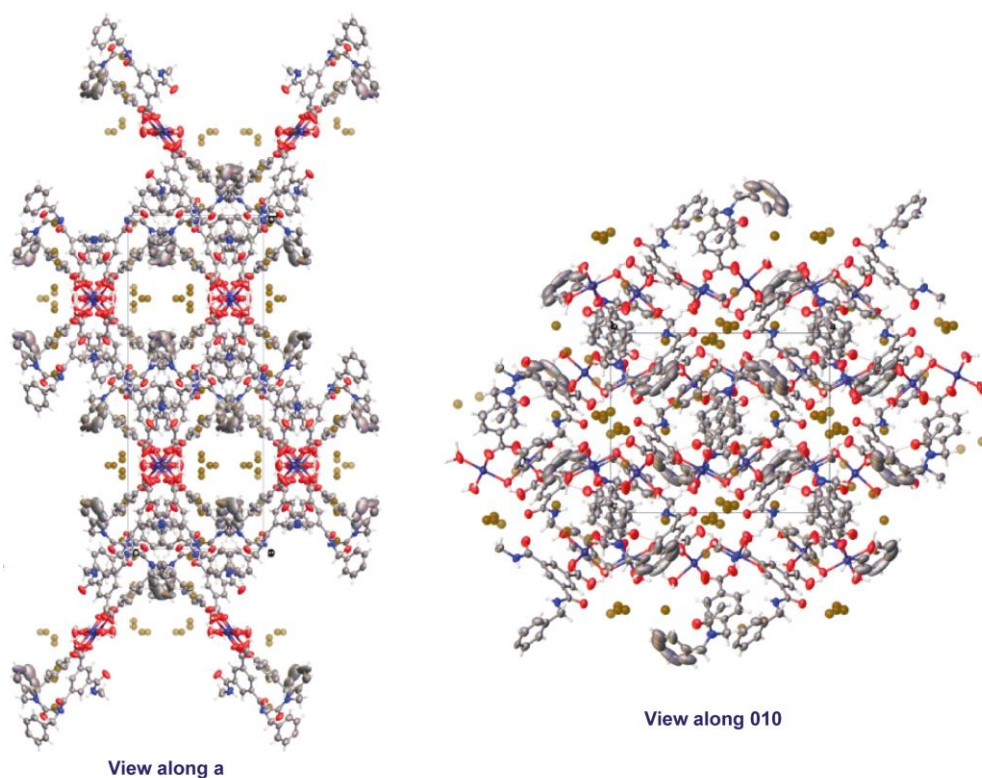


Figure S14. Single crystal X-ray Diffraction structure of evacuated crystals of **UMUMOF-8** showing the growth of asymmetric units along different views. Electron density of remaining non-evacuated solvent molecules is shown in brown.

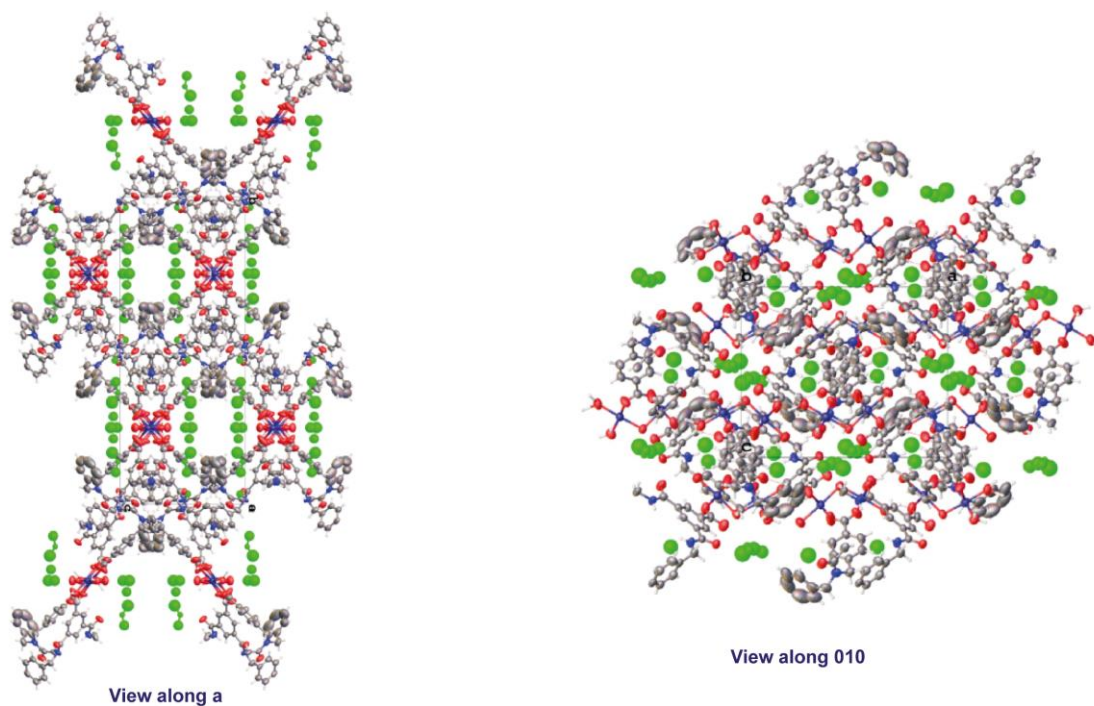


Figure S15. Single crystal X-ray Diffraction structure of **UMUMOF-8** crystals embedded in ethanol showing the growth of asymmetric units along different views. Electron density of ethanol molecules is shown in green.

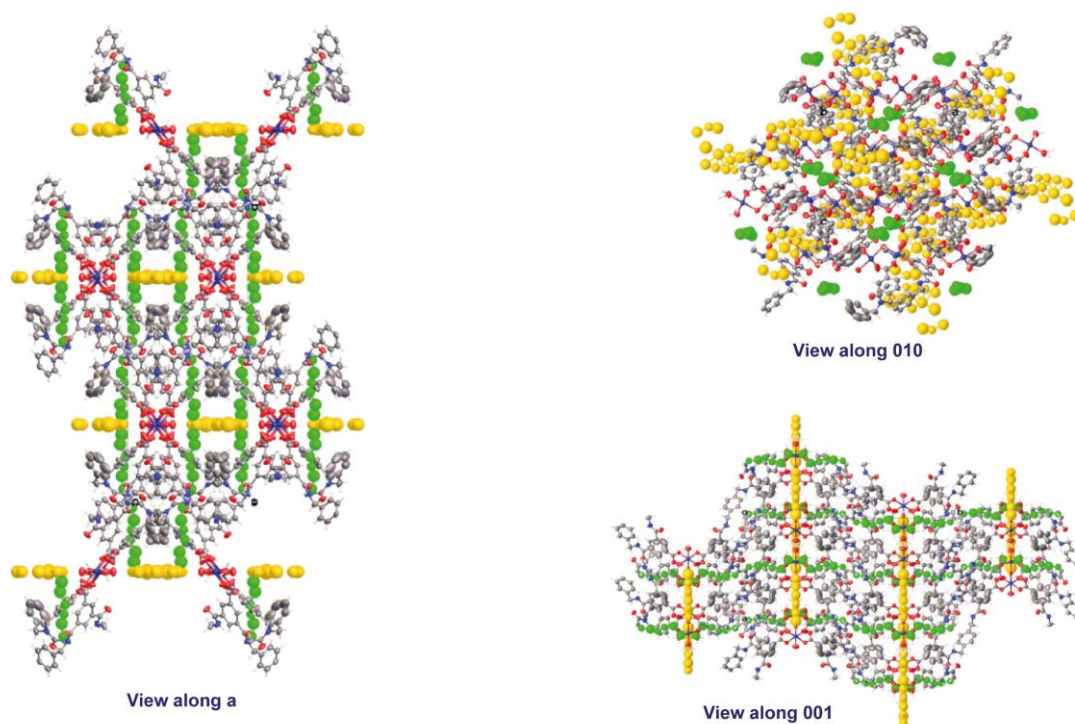


Figure S16. Single crystal X-ray Diffraction structure of **UMUMOF-8** crystals embedded in toluene showing the growth of asymmetric units along different views. Electron density of ethanol molecules is shown in green and that of toluene in yellow.

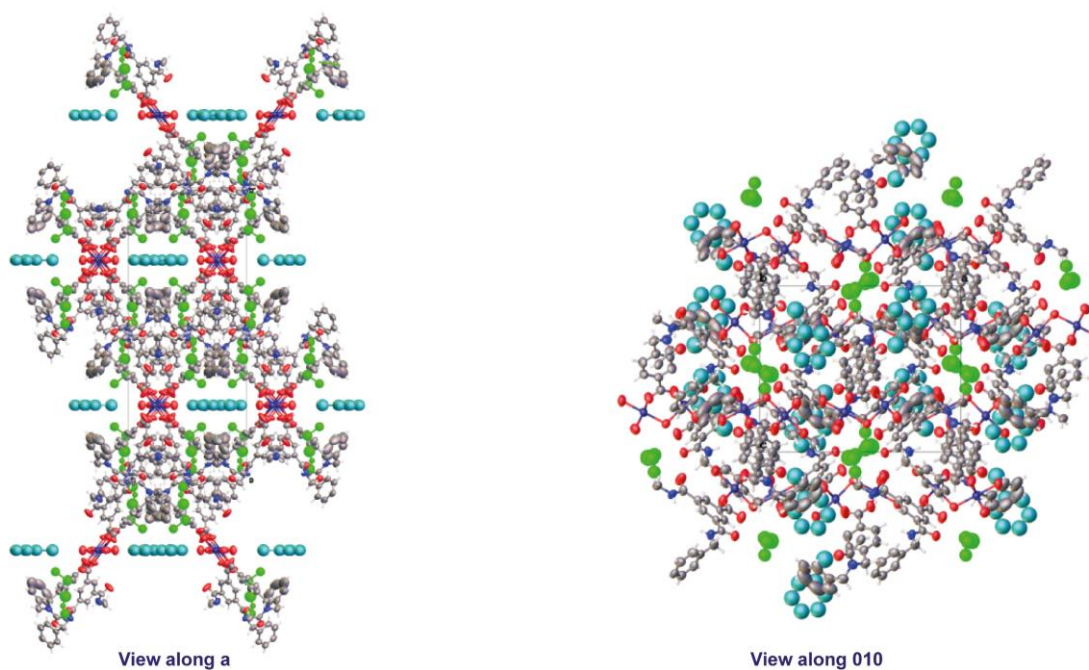
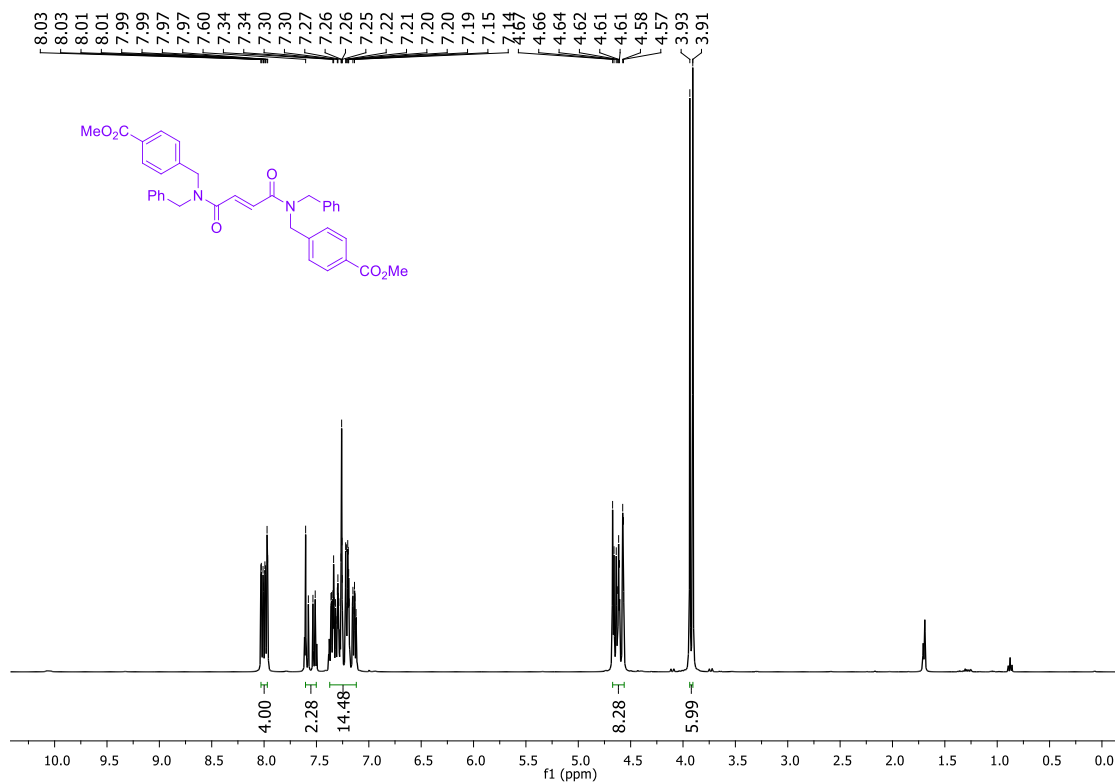


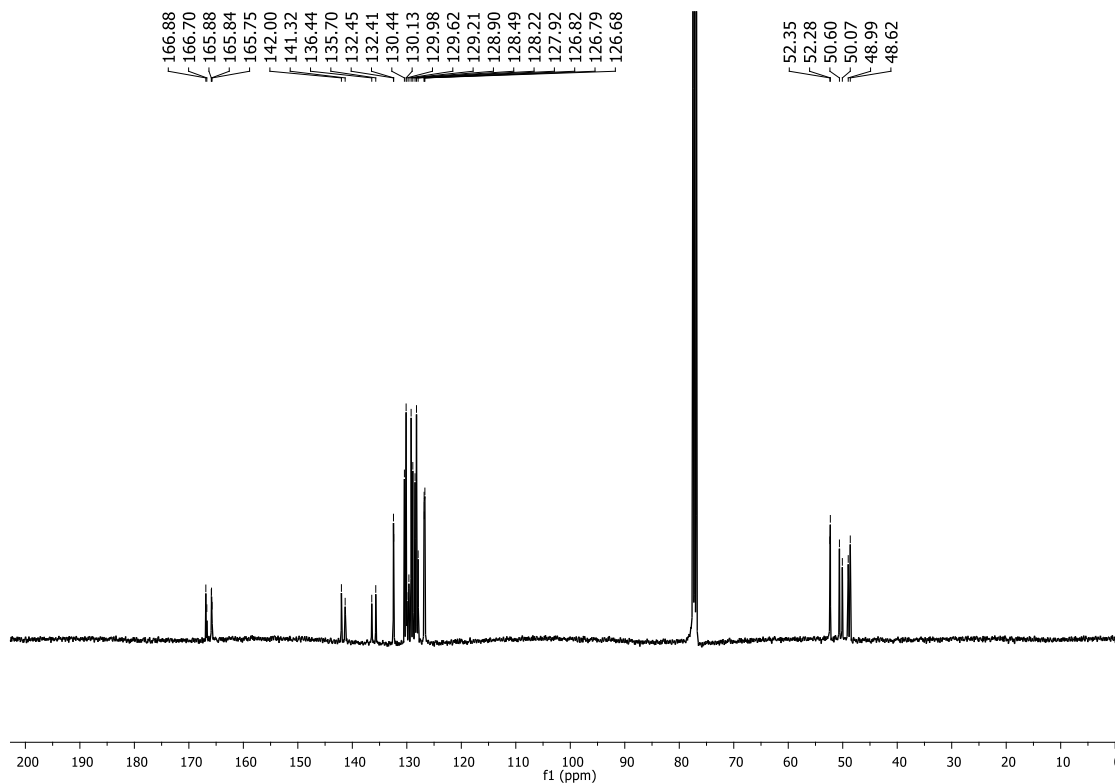
Figure S17. Single crystal X-ray Diffraction structure of **UMUMOF-8** crystals embedded in bromobenzene showing the growth of asymmetric units along different views. Electron density of ethanol molecules is shown in green and that of bromobenzene in blue.

11. ^1H and ^{13}C NMR spectra

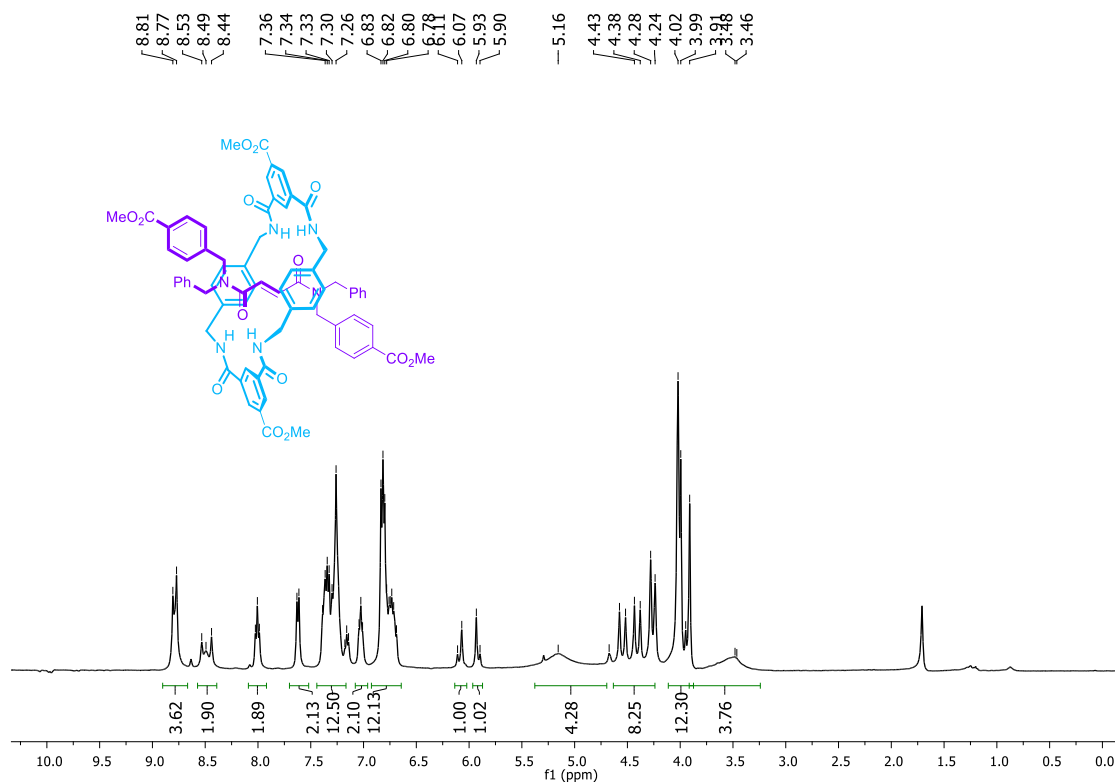
S1 (^1H NMR, 400 MHz, CDCl_3 , 298 K)



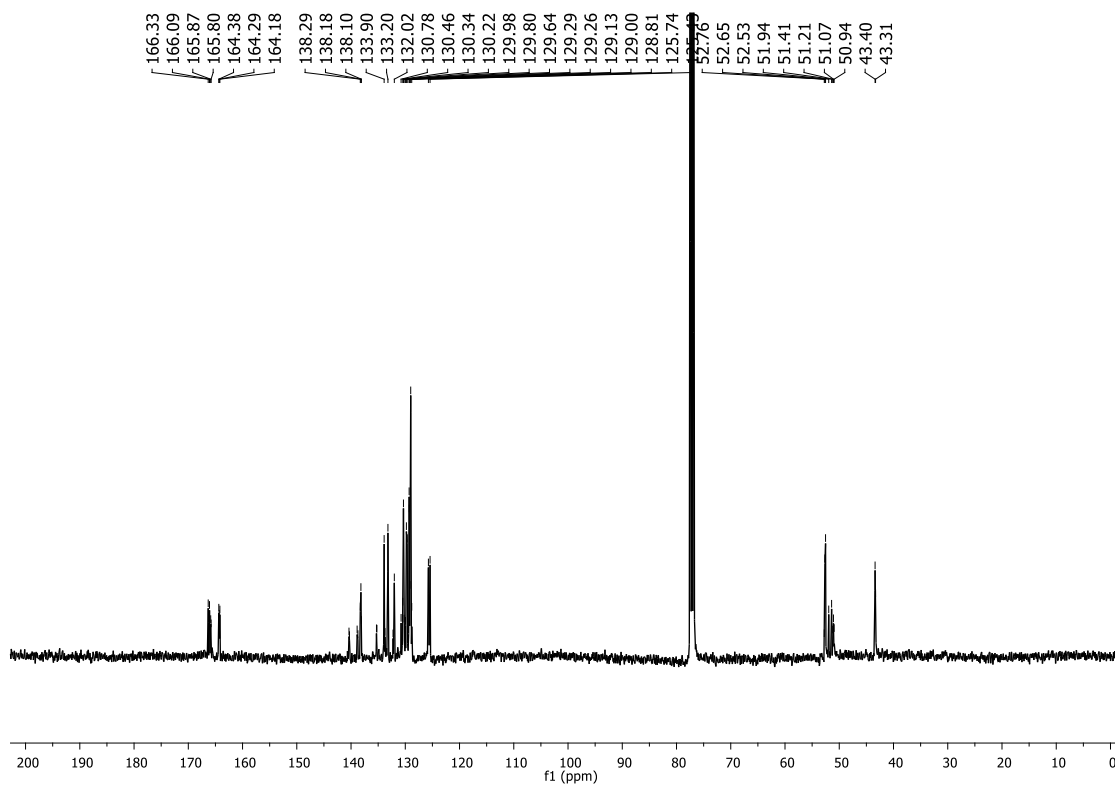
S1 (^{13}C NMR, 101 MHz, CDCl_3 , 298 K)



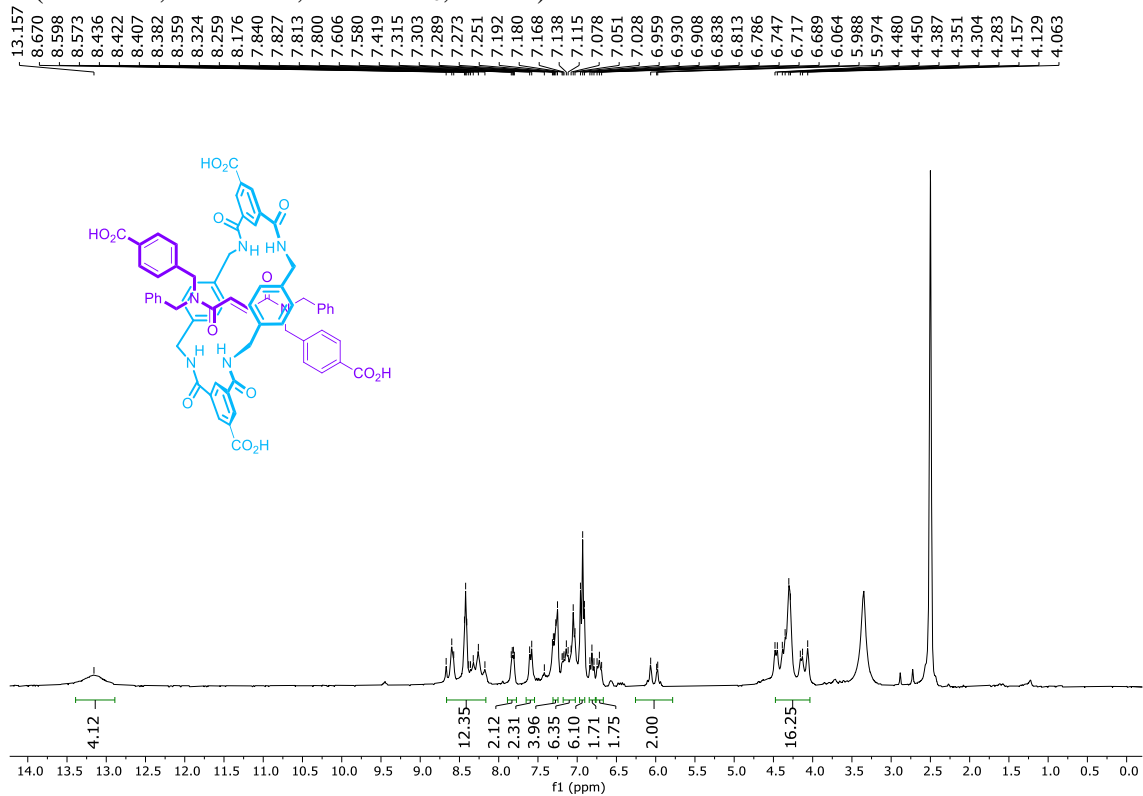
S2 (¹H NMR, 400 MHz, CDCl₃, 298 K)



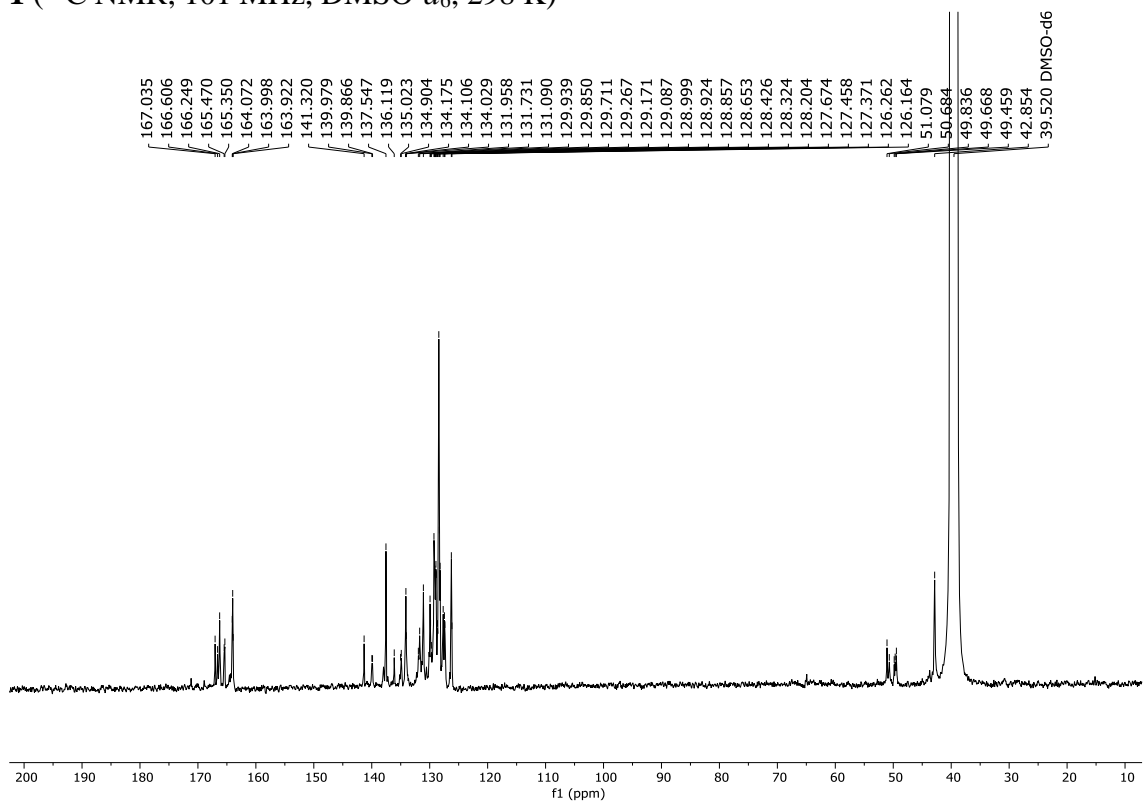
S2 (¹³C NMR, 101 MHz, CDCl₃, 298 K)



1 (^1H NMR, 400 MHz, $\text{DMSO-}d_6$, 298 K)



1 (^{13}C NMR, 101 MHz, $\text{DMSO-}d_6$, 298 K)



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