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VIP Metal–Organic Frameworks

A Catenated Strut in a Catenated Metal–Organic Framework**

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The integration of mechanically interlocked molecules (MIMs)[1] into metal–organic frameworks (MOFs)[2] provides a way of coupling[3] the workings[4] and addressability[5] of the former to the robustness and modularity of the latter. When MIMs are covalently linked to MOFs in such a well-defined fashion, the incoherent translational motion, which is observed in solution for the MIM progenitors, is eliminated. Although we have already shown that struts (2 nm in length) bearing components of MIMs, such as crown ethers,[6] pseudorotaxanes,[7] and catenanes,[8] can be reticulated into open MOFs,[9] when we attempted to build MOFs with three-dimensional structures incorporating catenane struts at this length scale, the only structure obtained[9b] was a two-dimensional one. Herein, we report the synthesis of a strut of exceptional length (3.3 nm) and describe its assembly into a three-dimensional MOF structure with vast openness, allowing bulky MIMs to be anchored at precise locations and with uniform relative orientations throughout the framework as a whole.

Specifically, a catenated dicarboxylic acid (CATDC) strut was joined to copper(I) to give a catenated three-dimensional MOF, namely MOF-1030, in which each unit cell contains a total of 7524 atoms (ignoring anions and solvent molecules) including those present in the 36 donor–acceptor [2]catenanes. While CATDC constitutes the longest link to be employed in MOF chemistry to date, the new extended structure is unique in so far as catenation is expressed simultaneously within the struts and the framework.

The synthesis of the extended strut CATDC, wherein 1,5-naphthoparaphenylene[36]crown-10 (NPP36C10) is grafted into its midriff by a modular synthetic protocol,[10] prior to being catenated with a cyclobis(paraquat-*p*-phenylene) (CBPQT^{4+}) ring,[11] is illustrated in Figure 1. $\mathrm{I_2}$-NPP36C10 was reacted in DMF employing Sonogashira coupling[12] conditions, with the substituted acetylene derivatives **1** (R = Me) and **2** (R = *t*Bu) to give, respectively, the struts CEME and CEBU.[13] De-esterification of CEME (base-promoted) and CEBU (acid-catalyzed) both afforded the same CEDC strut terminated by carboxyl groups. Although catenation with the well-established precursors of the CBPQT^{4+} ring, followed by counterion exchange, yielded the required CATDC for MOF preparation, this compound exhibited low solubility in most polar organic solvents and was therefore difficult to characterize. Consequently, CEME was also subjected to catenation under exactly the same conditions to yield the dimethyl diester CATME, which was fully characterized by ^{1}H NMR spectroscopy in solution and by X-ray crystallography in the solid state (see Supporting Information).

The crystal structure of CATME[13] is illustrated in Figure 2. In CATME, the NPP36C10 ring is endowed with planar chirality[14–16] as a consequence of the two pairs of *para*-oriented substituents on the hydroquinone ring. Racemization of the enantiomers is prevented on account of the lack of rotation of the central hydroquinone ring as a consequence of the length of the rigid strut. The planar chirality, which was revealed (see the Supporting Information, Figure S2) using a chiral shift reagent,[17] was confirmed by the existence of equal amounts of (*R*) and (*S*) enantiomers in the unit cell of CATME. In the solid state, the mechanically-interlocked components in the pendant [2]catenanes, namely the CBPQT^{4+} and NPP36C10 rings, adopt a geometry similar to that reported previously in the literature.[18] The linear rigid strut spans a distance of 32.9 Å in length between the two carboxylate carbon atoms. Overall, the incorporation of the pendant [2]catenane and the rigid strut renders CATDC, with its 120 non-hydrogen atoms (excluding counterions) and $M = 1595.82\ \mathrm{g\,mol^{-1}}$, the pinnacle in terms of complexity and size of links exploited in MOF synthesis to date.

MOF-1030 (Figure 3a) can be prepared from CATDC with $\mathrm{Cu(NO_3)_2{\cdot}2.5\,H_2O}$ employing reaction conditions (see Experimental Section) identical to those used[9b] in the

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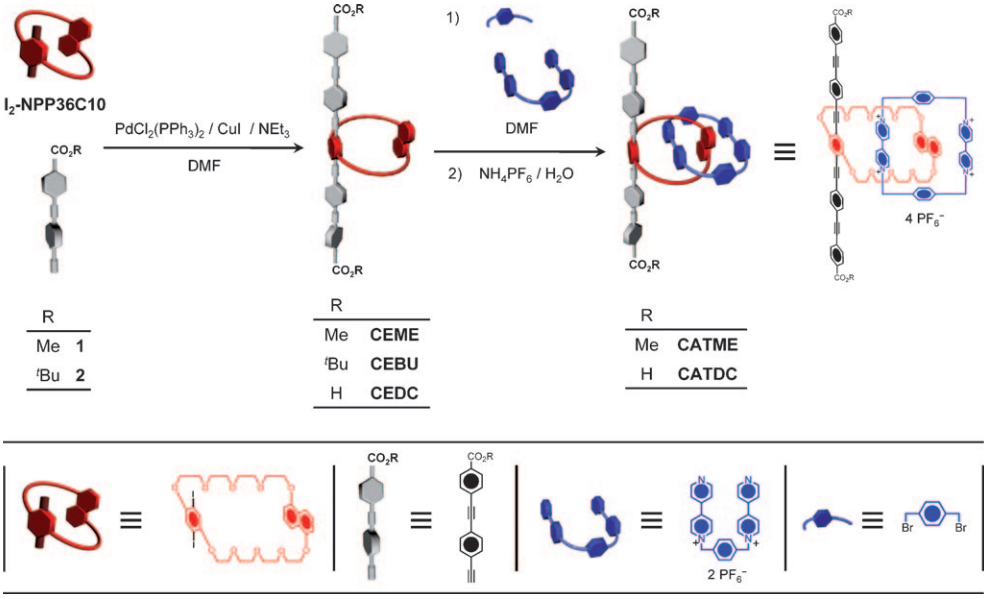

Figure 1. Catenated strut synthesis. CATME and CATDC were synthesized by the catenation of the CBPQT^{4+} ring around the 1,5-dioxynaphthalene unit in CEME and CEDC. CEDC was obtained from the de-esterification of CEBU (in TFA/MeCN) or CEME (in KOH/MeOH/CH$_2$Cl$_2$). Structural formulae defining the graphical representations are illustrated at the bottom.

synthesis of MOF-1011. Single-crystal X-ray diffraction reveals that MOF-1030[13] crystallizes in the trigonal space group $R\bar{3}c$, with unit cell parameters $a = b = 34.67$ Å, and $c = 98.98$ Å. In MOF-1030, each reduced copper(I) ion is bonded to two carboxylate groups from two struts and to one acetylenic bond from a third strut in an η^2 fashion (Figure 3b), after the same coordination as that present in MOF-1011. A closer examination of the strut in MOF-1030 reveals that the two carboxylate planes are twisted relative to each other. The π-alkyne copper(I) coordination and the length of the strut cause the π-conjugated system in the backbone to deteriorate, making the phenylene ring at one end (ring 1) almost perpendicular to the other phenylene rings (Figure 3a).

From a topological point of view, Cu(C≡C)(COO)$_2$ serves as a secondary building unit (SBU) with square-planar geometry. The points of extension of this square planar joint are two carbon atoms from the carboxylates and two carbon atoms connected to the acetylenic bond (Figure 3b). Meanwhile, strut CATDC can be divided into two different types of ditopic links, with lengths of 22.6 and 16.4 Å, corresponding to the distances between copper atoms

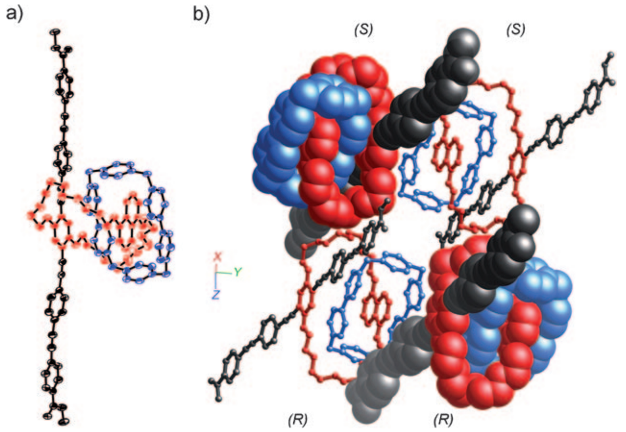

Figure 2. Crystal structure of CATME. a) ORTEP drawing of CATME, excluding hydrogen atoms and counterions. All ellipsoids are set at 80% probability. b) Packing of CATME in the solid state, with two catenanes illustrated by means of a space-filling representation. All the phenylene rings in the strut backbone are almost co-planar. The planar chirality of each catenane strut is labeled either (*R*) or (*S*). NPP36C10: red; CBPQT^{4+}: blue; linear strut: black. All hydrogen atoms and anions have been omitted for clarity.

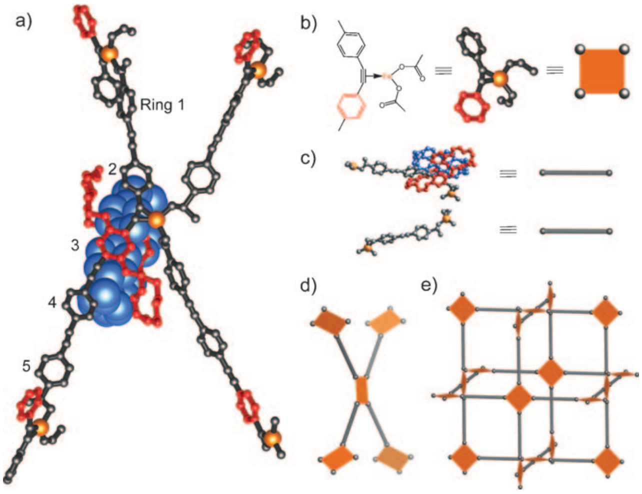

Figure 3. Assembly of MOF-1030 from square-planar joints and linear links. a) Crystal structure of MOF-1030. Ring 1 at one end of the catenane link is almost perpendicular to the other phenylene rings in the strut. b) Left: Each Cu$^+$ ion is bonded to two carboxylate groups from two struts and to one acetylenic bond from a third strut in an η^2 fashion. Center: This square-planar joint is shown in a ball-and-stick representation with the Cu$^+$ ion highlighted in orange. Right: The points of extension of this square planar joint are two carbon atoms from carboxylates and two carbon atoms connecting to the acetylenic group. c) Strut CATDC can be divided into two different types of ditopic links, connecting to square planar joints at both ends. d) Graphical representation of (a) with squares replacing the inorganic SBU, and rods replacing the organic links. The twist of Ring 1 results in neighboring square-planar joints being almost perpendicular to each other. e) The three-dimensional network with **nbo** topology was constructed by using squares and linear links. Organic struts: gray; crown ethers: red; CBPQT^{4+}: blue; Cu: orange spheres; inorganic SBUs: orange squares. All hydrogen atoms and anions have been omitted for clarity.

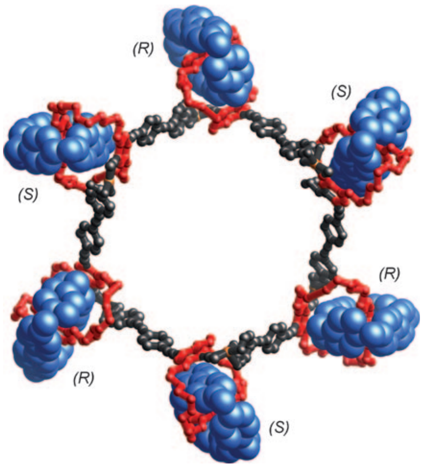

Figure 4. One of the hexagonal faces in MOF-1030. Hexagons are formed with six Cu^{+} ions as vertices. A ball-and-stick representation of a hexagonal array of catenanes with six of them inserted into the six edges. Organic struts: gray; crown ethers: red; CBPQT^{4+}: blue; Cu: orange. All hydrogen atoms and anions have been omitted for clarity.

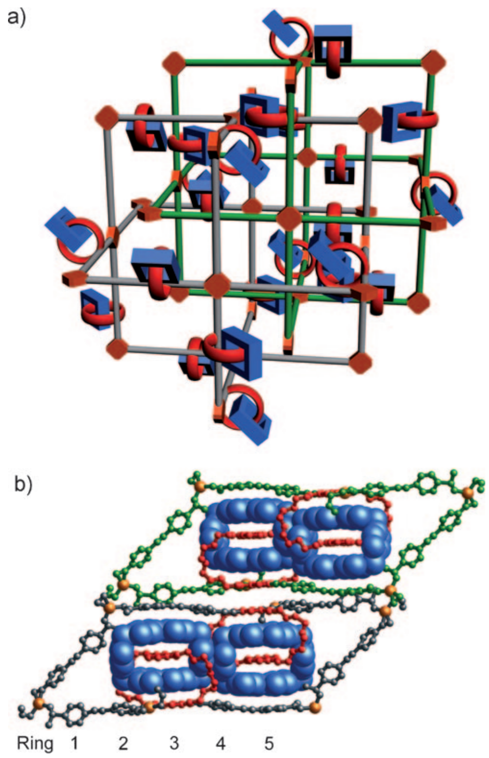

Figure 5. Catenated framework of MOF-1030. a) A representation of MOF-1030, which has a catenaned extended structure with the **nbo** topology in which the catenanes in the struts are located at precise locations and with uniform relative orientations. The two nets are shown in gray and green. Inorganic SBUs: orange squares; crown ethers: red rings; CBPQT^{4+}: blue square rings. b) In MOF-1030, two kinds of $\pi\cdots\pi$ stacking interactions exist in the frameworks: i) between CBPQT^{4+} cyclophanes (blue) and framework backbones (gray), and ii) between framework backbones from two nets (gray and green). Copper atoms: orange spheres; crown ethers: red spheres.

(Figure 3c). The twist of the carboxylate planes in the struts renders all the neighboring squares more or less perpendicular to each other (Figure 3d), leading to the observed three-dimensional structure.[19] This unique arrangement means that MOF-1030 has a network with an **nbo** topology,[20] assuming that both links are identical in terms of connectivity (Figure 3e).

The exceptional length of the struts provides vast openness to accommodate the bulky MIMs in the framework. Figure 4 illustrates a face in the **nbo** network of MOF-1030. Six donor–acceptor [2]catenanes point outward from the six links of the hexagon at precise locations and with uniform relative orientations. The (*R*) and (*S*) enantiomeric struts are present in a 1:1 ratio in the crystal structure of MOF-1030.

Considering the slender nature of the strut, it is not surprising that the backbone of MOF-1030 adopts a catenated framework (Figure 5a). The two nets are related by an inversion operation, and the closest copper atoms in two different nets are separated by 4.9 Å. Two kinds of $\pi\cdots\pi$ stacking interactions exist (Figure 5b) in the framework: i) between the CBPQT^{4+} rings and the framework backbones within which they are anchored, and ii) between the framework backbones from two catenated nets. $\pi\cdots\pi$ stacking between a CBPQT^{4+} ring and the neighboring strut backbone might explain why ring 2 is co-planar with the rings from 3 to 5, while ring 1 is not involved in stacking, and thus can be twisted, giving rise to the **nbo** topology network.

In summary, MOF-1030 (Figure 6) constitutes an example of the ordering of MIMs within a well-defined three-dimensional solid matrix. The catenated MOF-1030 brings the

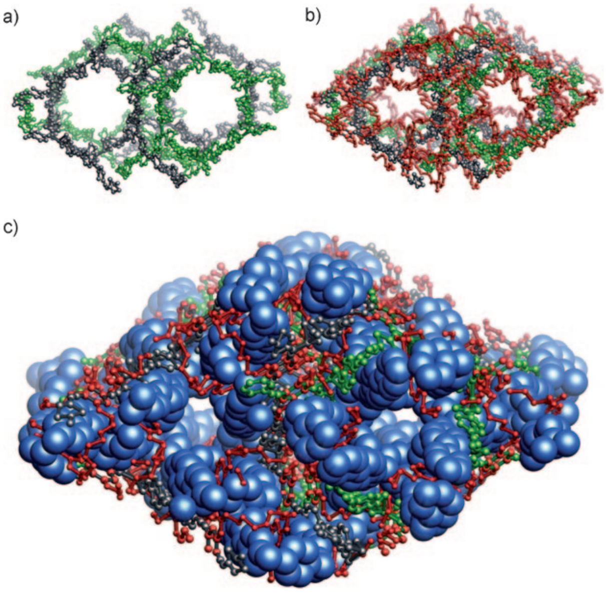

Figure 6. Catenated struts in catenated MOF-1030. a) View of MOF-1030 along the *c* axis. Crown ethers and CBPQT^{4+} rings are omitted. MOF-1030 is a catenated structure with two different frameworks shown in two different colors (gray and green). b) The framework with crown ethers represented in red. Hydrogen atoms, anions, and CBPQT^{4+} rings are omitted. c) Two interwoven nets, one in gray and the other in green, with the crown ethers shown in red and the CBPQT^{4+} rings in blue and featured in a space-filling manner.

prototypically active switching gear and machinery in the catenated struts to a standstill—at least as far as translational motion in three-dimensional space is concerned. Compared to the analogous pendant poly[2]catenanes,[21] MOFs made from catenated links significantly enhance the order and coherence of MIMs, with the potential to achieve mechanical movements in porous solid-state materials and hence ultimately switches organized in three-dimensional arrays for the next generation of functional materials directed towards ultrahigh-density electronics applications.

Experimental Section

MOF-1030: A solid mixture of CATDC (10.00 mg, 4.60×10^{-3} mmol; see Supporting Information) and $Cu(NO_3)_2\cdot2.5\,H_2O$ (20.00 mg, 8.60×10^{-2} mmol; Aldrich Chemical Co.) was dissolved in DMF (375 μL), EtOH (375 μL), and H_2O (250 μL) in a 4 mL vial. The vial was capped and placed in an isothermal oven at 85 °C for 6 days, after which time it was removed from the oven and allowed to cool to room temperature. A mixture of green and brown crystals was obtained. After decanting mother liquor, DMF (1.0 mL) and $CHBr_3$ (1.0 mL) was added to the vial. Because of the density difference of the two types of crystals, the brown crystals float to the top of the solution, while the green crystals stay at the bottom. Crystals of MOF-1030, which is the brown compound, were collected and rinsed with DMF (4×1.0 mL).

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[13] Crystallographic data for CEBU: $C_{78}H_{81}N_2O_{14}$, $M_r=1270.45$, triclinic, space group $P\bar{1}$, $a=10.8058(17)$, $b=11.873(3)$, $c=27.000(5)$ Å, $\alpha=92.247(16)$, $\beta=90.886(13)$, $\gamma=91.194(14)°$, $V=3460.3(11)$ Å^3, $D_{calcd}=1.219\ \mathrm{g\,cm^{-3}}$, $\lambda=1.54178$ Å, $Z=2$, R_1 $[I>2\sigma(I)]=0.0815$, wR_2 (all data) $=0.2222$, $GOF=1.013$. Crystallographic data for CATME: $C_{124}H_{124}N_{14}O_{14}P_4F_{24}$, $M_r=$

2614.25, triclinic, space group $P\bar{1}$, $a = 12.9773(2)$, $b = 13.7430(3)$, $c = 35.9341(6)$ Å, $\alpha = 79.1720(10)$, $\beta = 87.2080(10)$, $\gamma = 80.0320(10)°$, $V = 6198.7(2)$ Å^3, $D_{calcd} = 1.401$ g cm^{-3}, $\lambda = 1.54178$ Å, $Z = 2$, R_1 $[I > 2\sigma(I)] = 0.0491$, wR_2 (all data) = 0.1617, $GOF = 1.074$. Single crystal data for MOF-1030 after SQUEEZE: $C_{102}H_{88}Cu_1N_4O_{14}$, $M_r = 1657.30$, trigonal, space group $R\bar{3}c$, $a = b = 34.6678(5)$, $c = 98.979(3)$ Å, $V = 103021(4)$ Å^3, $D_{calcd} = 0.962$ g cm^{-3}, $\lambda = 1.54178$ Å, $Z = 36$, R_1 $[I > 2\sigma(I)] = 0.0980$, wR_2 (all data) = 0.3054, $GOF = 1.027$. CCDC 778456–778459 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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[16] The (*R*) and (*S*) enantiomer assignments of similar systems in our previous publications (Figures 5–8 in Ref. [10] and Figure 1 in Ref. [9b]) were incorrectly placed.

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