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# Water vapour induced reversible switching between a 1-D coordination polymer and a 0-D aqua complex†

Min Deng,<sup>‡a</sup> Soumya Mukherjee,<sup>‡b</sup> Yu-Jie Liang,<sup>a</sup> Xiao-Dan Fang,<sup>a</sup> Ai-Xin Zhu<sup>\*a</sup> and Michael J. Zaworotko<sup>‡b</sup>

**[Zn(3-tba)<sub>2</sub>], 1, a 1-D coordination polymer synthesised as 1 DMA, 1 $\alpha$ , transformed to a nonporous form, 1 $\beta$ , upon activation. 1 $\beta$  underwent further transformation to the dimeric complex [Zn(3-tba)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>], 2, above 40% RH. The reverse transformations, 2 to 1 $\beta$  and 1 $\beta$  to 1 $\alpha$ , were accomplished by heating and exposure to DMA, respectively, and were single-crystal-to-single-crystal phase changes. Single crystal X-ray diffraction revealed that the second transformation resulted from Zn–carboxylate bond breakage and concomitant coordination of water molecules. Other solvent molecules did not induce a phase change.**

Metal–organic materials (MOMs),<sup>1</sup> especially porous MOMs such as metal–organic frameworks (MOFs)<sup>2</sup> and porous coordination polymers (PCPs),<sup>3</sup> have received considerable attention with respect to their gas and vapour adsorption properties.<sup>4</sup> Whereas > 100 000 coordination networks have been deposited in the MOF subset of the Cambridge Structural Database (CSD),<sup>5</sup> only a small proportion, < 100, are known to exhibit type F-IV isotherms with reversible transformations between nonporous (closed) and porous (open) phases.<sup>6</sup> Such stepped isotherms are of topical interest because their “switching” between closed and open phases can result in relatively high uptake capacity and, perhaps counter-intuitively, stronger separation selectivity than rigid porous materials with similar pore size.<sup>7,8</sup> Indeed, benchmark binding to C<sub>2</sub>H<sub>2</sub> has been observed for a switching sorbent through an induced-fit mechanism reminiscent of enzyme–substrate binding.<sup>9</sup>

Single-crystal-to-single-crystal (SCSC) transformations in switching sorbents can provide insight into structure–property

relationships through single-crystal X-ray diffraction (SCXRD).<sup>10</sup> SCSC transformations can typically be induced by external stimuli, e.g. gas/vapour uptake and/or removal,<sup>11,12</sup> temperature change,<sup>13</sup> pH change,<sup>14</sup> light<sup>15</sup> and cation or anion exchange.<sup>16,17</sup> Water vapour sorption is of particular relevance as it can bind with open metal centres<sup>18,19</sup> or physisorb, thereby being relevant to applications such as atmospheric water harvesting and dehumidification. Most MOMs exhibit type F-IV water vapour isotherms that result from pore filling (capillary condensation, Table S6 in ESI†).<sup>20</sup> Our literature survey revealed only 12 MOMs (Table S6, ESI†) that exhibit a type F-IV stepped isotherm caused by water-induced structural changes and, to our knowledge, water-induced 1D  $\rightarrow$  0D SCSC structural phase changes with a single-step are unstudied. Such transformations have the potential to exhibit high selectivity for water over alcohols. Indeed, some examples display selective water uptake over alcohols attributed to structural transformations.<sup>19a,21</sup>

Herein, we report that the new 1D coordination polymer [Zn(3-tba)<sub>2</sub>]-DMA (1 $\alpha$ ; 3-Htba = 3-(4H-1,2,4-triazol-4-yl)benzoic acid; DMA = *N,N*-dimethylacetamide), 1 $\alpha$ , underwent SCSC transformation to 1 $\beta$ , a nonporous phase, upon removal of DMA. 1 $\beta$  in turn transformed to a discrete, binuclear complex, 2, upon exposure to water vapour. Insight into these reversible transformations comes from the results of SCXRD studies.

Solvothermal reaction of 3-Htba with Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O in DMA at 105 °C afforded diamond-shaped crystals of 1 $\alpha$  (synthetic details are available in ESI†). SCXRD revealed that 1 $\alpha$  crystallized in the triclinic space group *P* $\bar{1}$  and that it displays a 1D chain structure with spiro linkages (Fig. 1). Zn(II) cations adopt a tetrahedral coordination geometry through two oxygen atoms from different 3-tba ligands and two nitrogen atoms from two additional 3-tba ligands. Pairs of 3-tba ligands serve as V-shaped linkers between adjacent Zn(II) cations to form [Zn<sub>2</sub>(3-tba)<sub>2</sub>] rings that are further connected into a one-dimensional (1D) coordination polymer (Fig. 1). Adjacent chains are cross-linked by C–H $\cdots$ O interactions (C $\cdots$ O = 3.049(6)–3.097(6) Å, H $\cdots$ O = 2.39–2.44 Å, C–H $\cdots$ O = 127–128°) to form a supramolecular layer (Fig. S1, ESI†).  $\pi$ – $\pi$  and C–H $\cdots$  $\pi$

<sup>a</sup> Faculty of Chemistry and Chemical Engineering, Yunnan Normal University, Kunming, 650500, China. E-mail: zaxchem@126.com

<sup>b</sup> Department of Chemical Sciences, Bernal Institute, University of Limerick, Limerick, V94T9PX, Ireland. E-mail: xtal@ul.ie

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‡ These authors contributed equally to this work.



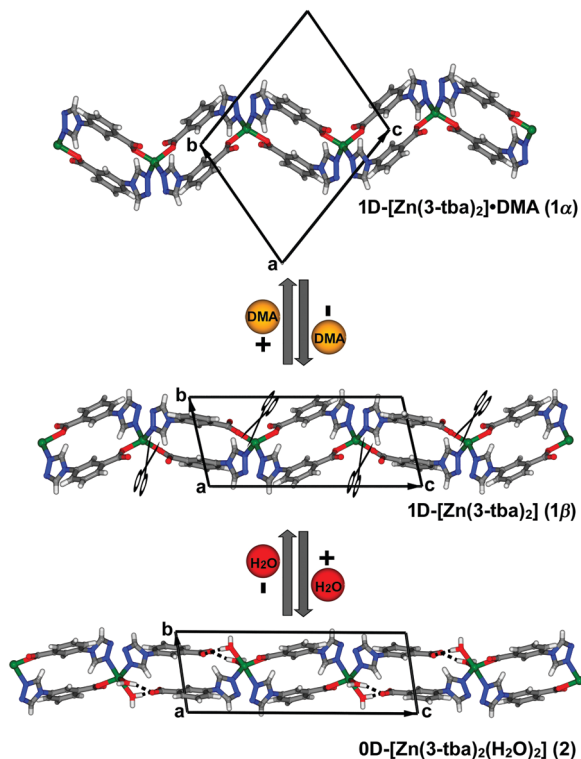


Fig. 1 Structural transformations in  $[\text{Zn}(\text{3-tba})_2]$  involving **1 $\alpha$** , **1 $\beta$**  and **2** (unit cells are illustrated). Charge-assisted H-bonding (black broken lines) occurs between aqua ligands and free carboxylate moieties in **2**.

interactions between 1D chains also stabilise the supramolecular layer (Fig. S2, ESI<sup>†</sup>) which pack through C–H $\cdots$ N interactions (C $\cdots$ N = 3.486(6) Å, H $\cdots$ N = 2.57 Å, C–H $\cdots$ N = 168°) between layers (Fig. S3, ESI<sup>†</sup>). Along the *a*-axis, the crystal packing of **1 $\alpha$**  results in 1D rhombic channels with an effective pore diameter of  $4.9 \times 7.3$  Å<sup>2</sup> (Fig. 2 and Fig. S4, ESI<sup>†</sup>). Void volume in the channel (310.3 Å<sup>3</sup>) is *ca.* 27.4% of the crystal volume (1134.2 Å<sup>3</sup>) which is occupied by DMA guest molecules. Thermogravimetric analysis (TGA) revealed that as-synthesized **1 $\alpha$**  loses guest molecules (obs. 16.11%, calc. 16.47%) from 78 °C to 180 °C and remains stable to 290 °C (Fig. S17, ESI<sup>†</sup>).

Heating **1 $\alpha$**  under vacuum at 150 °C overnight resulted in SCSC transformation to **1 $\beta$** . SCXRD revealed that the **1 $\beta$**  is a contorted version of **1 $\alpha$**  with the same connectivity. **1 $\beta$**  also crystallized in triclinic space group *P* $\bar{1}$  but with 24.8% shrinkage of its unit-cell volume relative to **1 $\alpha$** . PLATON calculations indicated that **1 $\beta$**  contains no residual solvent-accessible void and so it is nonporous (Fig. 2). TGA and FT-IR data support the guest-free nature of **1 $\beta$**  (Fig. S17 and S19, ESI<sup>†</sup>). Transformation between **1 $\alpha$**  and **1 $\beta$**  was accompanied by distortions of  $[\text{Zn}_2(\text{3-tba})_2]$  rings and contraction of interstitial spaces (Fig. S5, ESI<sup>†</sup>). Meanwhile, rotation of the 3-tba ligand and a hinge-like motion associated with carboxylate coordination occurred (Table S2, ESI<sup>†</sup>). Aromatic  $\pi$ – $\pi$  stacking interactions were found to be present in **1 $\beta$**  (Tables S4, S5 and Fig. S7, ESI<sup>†</sup>). The structural transformation associated with guest removal was found to be reversible as **1 $\beta$**  reverted to **1 $\alpha$**  after soaking in DMA at room temperature for 1 day

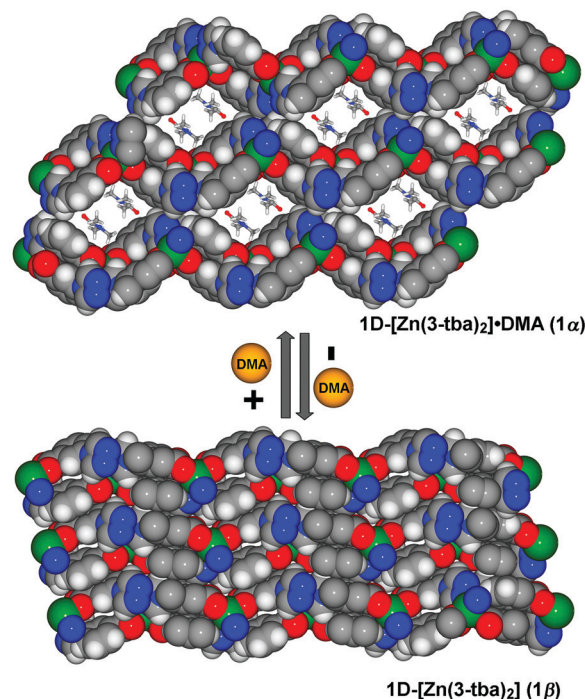


Fig. 2 Space-filling diagrams of the reversible structural transformation between **1 $\alpha$**  and **1 $\beta$**  (viewed along the *a*-axis, for more details see Fig. S5, ESI<sup>†</sup>).

(Fig. 3 and Fig. S9, ESI<sup>†</sup>). **1 $\beta$**  was observed to transform to a new phase, **2**, after exposure to humidity (Fig. S11 and S12, ESI<sup>†</sup>).

Single crystals of **2** were obtained after exposure of **1 $\beta$**  to water vapour (vial-in-vial method, details in the ESI<sup>†</sup>). Such SCSC transformations of low-dimensional coordination networks between multiple phases are relatively rare.<sup>22</sup> SCXRD revealed that the formation of **2**,  $[\text{Zn}(\text{3-tba})_2(\text{H}_2\text{O})_2]$ , a discrete complex, involved the following: (a) change from 1-D to 0-D dimensionality; (b) cleavage of some Zn–carboxylate coordination bonds; (c) insertion of coordinated water molecules that form hydrogen bonds to the uncoordinated carboxylate moieties; (d) change of the Zn coordination geometry from tetrahedral to trigonal bipyramidal. As revealed by Fig. 1, **2** is composed of Zn(II) ions that adopt a distorted trigonal bipyramidal coordination geometry ( $\tau = 0.77$ ) with two nitrogen atoms from two 3-tba ligands, one oxygen atom from another 3-tba ligand and two coordinated water molecules. Coordination of water molecules to Zn(II) in effect results in the insertion of water molecules into a Zn–carboxylate bond and formation of charge assisted hydrogen bonds (O $\cdots$ O = 2.535 and 2.626 Å, Fig. 1). Furthermore, the other hydrogen atom from one of the coordinated water molecules formed H-bonds (O $\cdots$ O = 2.737 Å) with uncoordinated O atoms of 3-tba ligands in adjacent complexes whereas the remaining hydrogen atom formed bifurcated H-bonds with two basic N atoms (O $\cdots$ N = 3.146 and 3.254 Å, Fig. S8, ESI<sup>†</sup>). The FT-IR spectrum of **2** indicates that C=O (1600 cm<sup>−1</sup>) and C–O ( $\sim$ 1051 cm<sup>−1</sup>) vibrations are different from **1 $\alpha$**  and **1 $\beta$** , consistent with the respective coordination environments (Fig. S19, ESI<sup>†</sup>). In contrast to **1 $\alpha$**  and



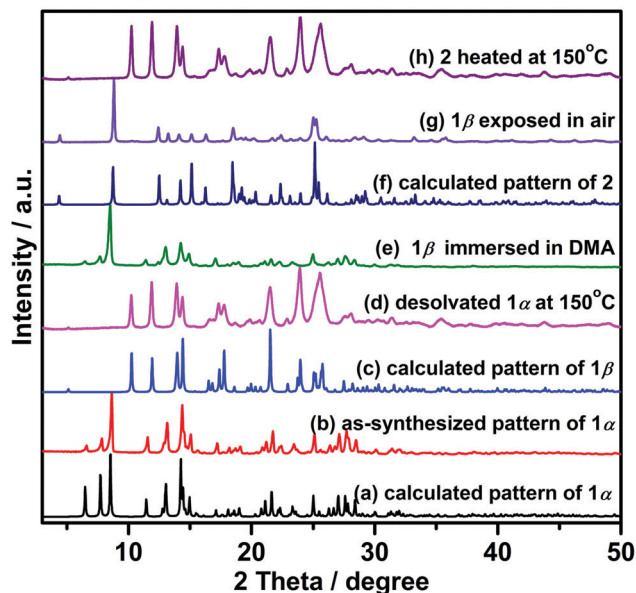


Fig. 3 PXRD patterns of (a) **1α** calculated from SCXRD data, (b) as-synthesized **1α** at room temperature, (c) **1β** calculated from SCXRD data, (d) desolvated **1α** at 150 °C, (e) **1β** immersed in DMA for 1 day, (f) calculated **2** from SCXRD data, (g) **1β** exposed to humid air (ca. 45% RH), and (h) dehydrated **2** at 150 °C.

**1β**, there are no aromatic  $\pi$ - $\pi$  stacking interactions between triazole rings (Table S4, ESI†) but multiple O-H...O and O-H...N hydrogen bonds formed between aqua ligands and uncoordinated N or O atoms of 3-tba ligands (Table S3 and Fig. S8, ESI†). **2** was found to reversibly revert to **1β** *in vacuo* at 150 °C (Fig. S10, ESI†). Attempts to obtain **2** by direct routes were unsuccessful.

Gas sorption experiments for N<sub>2</sub> at 77 K and CO<sub>2</sub> at 195 K were performed on **1β**, which exhibited a type II nitrogen adsorption isotherm (Fig. S20, ESI†) characteristic of a non-porous solid. The BET surface area was determined to be 5.6 m<sup>2</sup> g<sup>-1</sup>. The CO<sub>2</sub> adsorption isotherm collected at 195 K likewise indicated that **1β** is nonporous (Fig. S13 and S20, ESI†).

Vapour sorption isotherms of water, methanol and ethanol for **1β** were conducted at 298 K. As shown in Fig. 4, no water was adsorbed in the low humidity region but water uptake showed a sudden increase (step) at 46% RH. Such a profile is consistent with a structural transformation. The desorption isotherm exhibits large hysteresis, indicating strong sorbate-water interactions as would be expected from the crystal structure of **2**. To our knowledge, this is the first example of a water-induced 1D → 0D SCSC structural transformation with a one-step type F-IV adsorption isotherm (Table S6, ESI†). At 50% RH, the uptake reached 102 cm<sup>3</sup> g<sup>-1</sup>, corresponding to approximately 2.0 H<sub>2</sub>O molecules per Zn cation, a value in accordance with the TGA measured for **1β** exposed to water vapour for one day (Fig. S18, ESI†). As revealed by Fig. 4, methanol and ethanol adsorption exhibited no uptake up to  $P/P_0 = 0.96$ . To the best of our knowledge, only one example of a sorbent that displays selective water uptake over alcohols has been observed for materials with type F-IV isotherms.<sup>21a</sup>

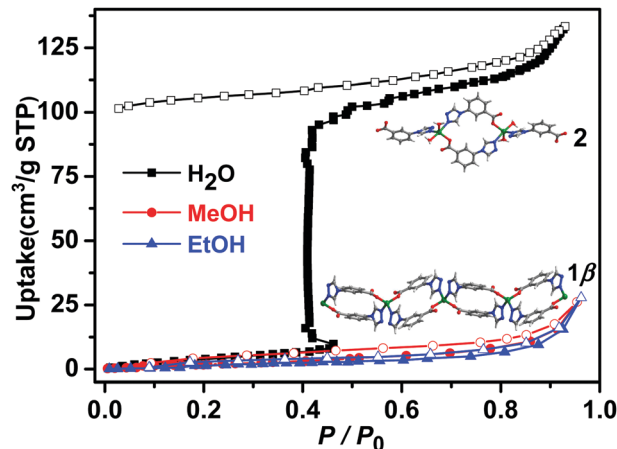


Fig. 4 Water, methanol and ethanol vapour sorption isotherms of **1β** collected at 298 K (closed and open symbols denote adsorption and desorption, respectively).

In **1β**, one Zn-carboxylate bond in each formula unit deviates from the carboxylate group plane (20.6°, Table S2 and Fig. S5, ESI†). The structure of **2** indicates that two water molecules had in effect inserted into a Zn-carboxylate bond, in turn forming two charge assisted OH...carboxylate H-bonds (Fig. 1). Such a motif has been observed in molecular crystals as exemplified by DL-tartaric acid monohydrate<sup>23</sup> and there are ca. 1400 hits in the CSD database for this motif. The coordination of water molecules and charge assisted OH...carboxylate hydrogen bonds supports this water-induced phase transformation. It seems unlikely that the structure of **2** would exist with MeOH or EtOH molecules as there is also H-bonding to the adjacent chains (Fig. S5 and S8, ESI†), perhaps explaining why MeOH and EtOH were not adsorbed.

The sorption isotherms of **1β** for acetonitrile, acetone and benzene also indicated no uptake (Fig. S22, ESI†) and PXRD data revealed that crystals of **1β** were unaffected by exposure to methanol, ethanol, acetonitrile, acetone or benzene vapours in contrast to water vapour, which induced transformation from **1β** to **2** (Fig. S14 and S15, ESI†). Not only was **1β** selective for water, but it was found to retain its water uptake after 5 consecutive activation-uptake cycles (Fig. 5 and Fig. S16, ESI†), indicating that **1β** is recyclable.

In summary, we report a new 1D coordination polymer, [Zn(3-tba)<sub>2</sub>] (**1α**), that transformed to **1β** upon removal of guests. **1β** further transformed to a 0D aqua complex [Zn(3-tba)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>], **2**, upon exposure to water vapour above 40% RH. Both transformations were verified by SCXRD studies and found to be reversible. Interestingly, **1β** exhibited a one-step type F-IV water adsorption isotherm concomitant with the 1D → 0D SCSC structural transformation. The coordination of two water molecules and self-assembly sustained by charge-assisted OH...carboxylate hydrogen bonds are likely key drivers for this switching event along with other H-bonds formed by the aqua ligands. The water-triggered transformation enabled selective and reversible adsorption of water vapour over other vapours such as methanol, ethanol, acetone, acetonitrile and



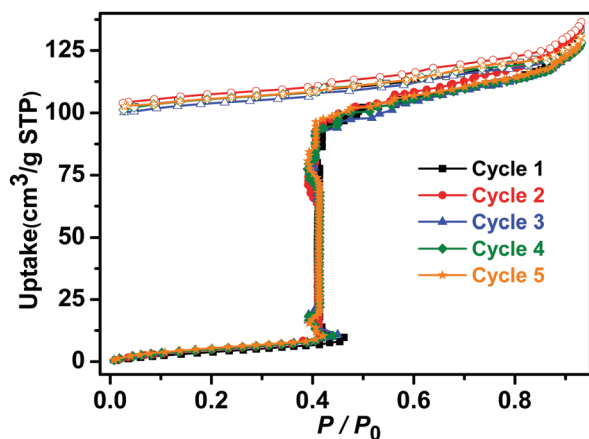


Fig. 5 Five consecutive activation–uptake cycles of water sorption isotherms of **1β** at 298 K (closed and open symbols denote adsorption and desorption, respectively).

benzene. Transformation between **1β** and **2** implies that there could be other low-dimensional MOMs or molecular compounds that might serve as switching water sorbents given that there are *ca.* 1400 crystal structures with diaqua-carboxylate motifs archived in the CSD.

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## Conflicts of interest

There are no conflicts to declare.

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