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

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**[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···O interactions (C···O = 3.049(6)–3.097(6) Å, H···O = 2.39–2.44 Å, C–H···O = 127–128°) to form a supramolecular layer (Fig. S1, ESI†).  $\pi$ – $\pi$  and C–H··· $\pi$

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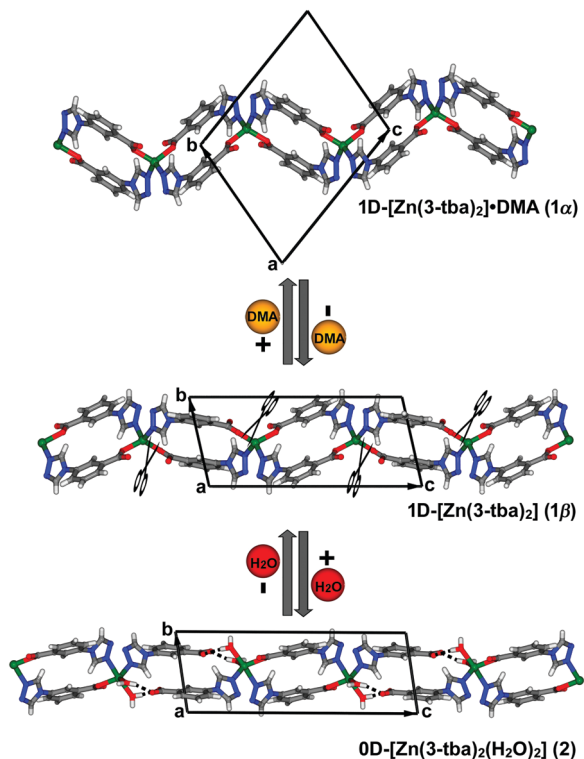


Fig. 1 Structural transformations in  $[\text{Zn}(\text{3-tba})_2]$  involving  $1\alpha$ ,  $1\beta$  and  $2$  (unit cells are illustrated). Charge-assisted H-bonding (black broken lines) occurs between aqua ligands and free carboxylate moieties in  $2$ .

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

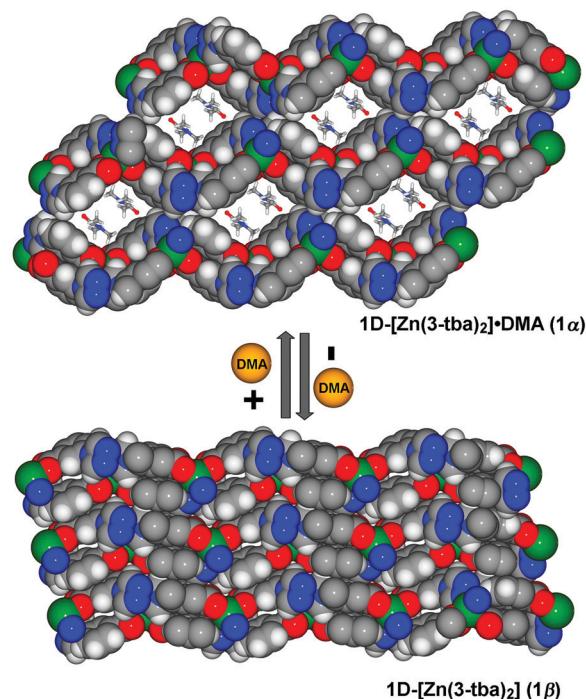


Fig. 2 Space-filling diagrams of the reversible structural transformation between  $1\alpha$  and  $1\beta$  (viewed along the *a*-axis, for more details see Fig. S5, ESI<sup>†</sup>).

(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



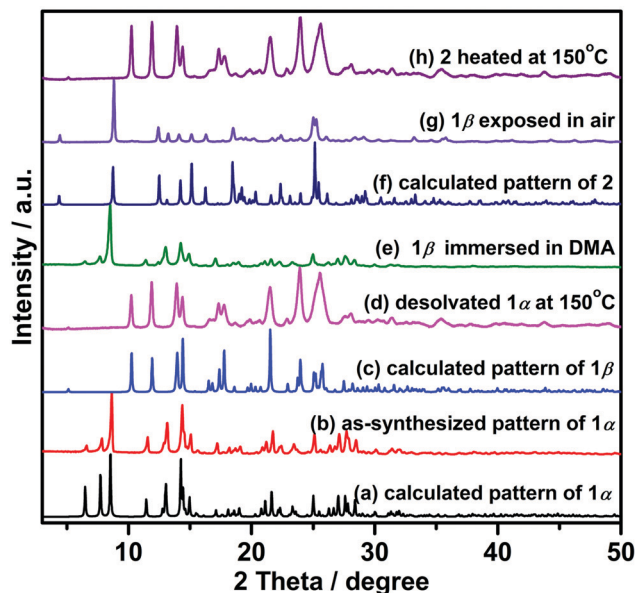


Fig. 3 PXRD patterns of (a)  $1\alpha$  calculated from SCXRD data, (b) as-synthesized  $1\alpha$  at room temperature, (c)  $1\beta$  calculated from SCXRD data, (d) desolvated  $1\alpha$  at 150 °C, (e)  $1\beta$  immersed in DMA for 1 day, (f) calculated  $2$  from SCXRD data, (g)  $1\beta$  exposed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

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

Gas sorption experiments for  $N_2$  at 77 K and  $CO_2$  at 195 K were performed on  $1\beta$ , which exhibited a type II nitrogen adsorption isotherm (Fig. S20, ESI<sup>†</sup>) 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  $CO_2$  adsorption isotherm collected at 195 K likewise indicated that  $1\beta$  is nonporous (Fig. S13 and S20, ESI<sup>†</sup>).

Vapour sorption isotherms of water, methanol and ethanol for  $1\beta$  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  $\rightarrow$  0D SCSC structural transformation with a one-step type F-IV adsorption isotherm (Table S6, ESI<sup>†</sup>). 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  $1\beta$  exposed to water vapour for one day (Fig. S18, ESI<sup>†</sup>). 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>

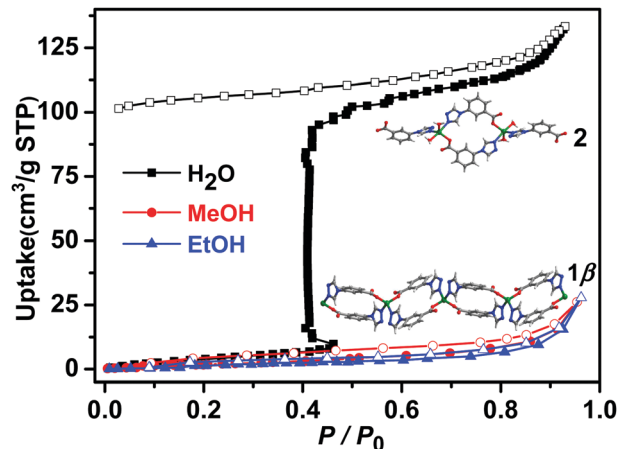


Fig. 4 Water, methanol and ethanol vapour sorption isotherms of  $1\beta$  collected at 298 K (closed and open symbols denote adsorption and desorption, respectively).

In  $1\beta$ , one Zn-carboxylate bond in each formula unit deviates from the carboxylate group plane (20.6°, Table S2 and Fig. S5, ESI<sup>†</sup>). 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 $\cdots$ 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 $\cdots$ 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<sup>†</sup>), perhaps explaining why MeOH and EtOH were not adsorbed.

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

In summary, we report a new 1D coordination polymer, [Zn(3-tba)<sub>2</sub>] ( $1\alpha$ ), that transformed to  $1\beta$  upon removal of guests.  $1\beta$  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,  $1\beta$  exhibited a one-step type F-IV water adsorption isotherm concomitant with the 1D  $\rightarrow$  0D SCSC structural transformation. The coordination of two water molecules and self-assembly sustained by charge-assisted OH $\cdots$ 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



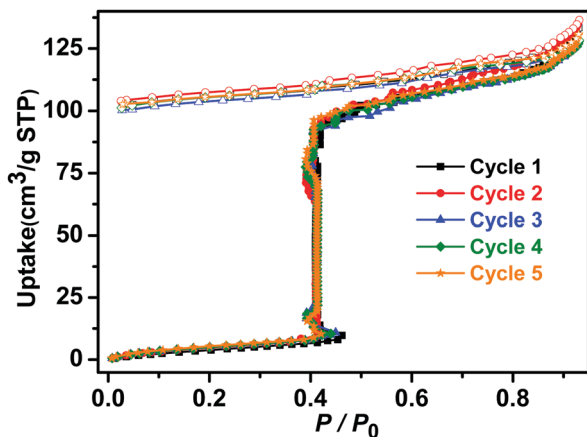


Fig. 5 Five consecutive activation-uptake cycles of water sorption isotherms of **1β** at 298 K (closed and open symbols denote adsorption and desorption, respectively).

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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