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Syntheses and Crystal Structures of Two Coordination Polymer Based on 1,3,5-Tris(Imidazol-1-Ylmethyl)-2,4,6-Trimethylbenzene[1]

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Abstract—Two novel coordination polymers, $[Zn_2(CAM)_2(Titmb)]$ (**I**) and $[Zn_4Co(OH)_2(BTEC)_2(Titmb)_2(H_2O)_2] \cdot 2H_2O$ (**II**) (H_2CAM = camphor acid, H_4BTEC = 1,2,4,5-benzenetetracarboxylic acid, and Titmb = 1,3,5-tris(imidazol-1-ylmethyl)-2,4,6-trimethylbenzene), have been hydrothermal prepared and characterized by IR spectroscopy, elemental analysis, and single-crystal X-ray diffraction. Complex **I** displays an unusual three-dimensional (3,4,6)-connected network with $(4.6^4.8)_2(4^2.6^4.8^9)(6^2.8)_2$ topology. Complex **II** exhibits an interesting heterometallic 3D metal-organic framework. Moreover, the luminescent properties of **I** have been investigated in the solid state at room temperature.

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INTRODUCTION

The design and construction of metal-organic coordination frameworks is of current interest in the fields of supramolecular chemistry and crystal engineering. The major reasons for this interest stem from their intriguing variety of topologies and structural diversity, such as helixes and diamondoid nets, and from their potential applications for heterogeneous catalysis, molecular recognition, magnetism, gas storage, ion exchange, nonlinear optics, and electrical conductivity [1–4]. In order to build these molecular architectures, polycarboxylates are often employed as bridging ligands to construct metal-organic coordination frameworks owing to their versatile coordination modes and high structural stability [5, 6]. MOFs, based on rigid carboxylate linkers, have been widely studied and highly explored [7]. In contrast to the rigid ligands, the rational design of coordination polymers based on flexible ligands are quite interesting in terms of the self-assembly process and structure-property relationship [8]. Because of the conformational freedom of the flexible ligands, it results in formation of both discrete macrocylces and infinite polymeric structures; conversion between these two types of structures (discrete and polymer) could also be achieved by the ring-opening isomerism [9]. Cao and his group studied and reviewed the coordination polymers based on flexible ligands [10]. The conformational freedom of flexible ligands offers the possibility to construct unpredictable and interesting coordination networks with useful properties. The final structures, based on flexible ligands, are subjected to several factors, such as synthetic conditions (temperature, pH, pressure, and solvents), coordination geometry of metal ions, geometrical disposition of donor sites, template molecules, and so on [11–13]. Thus, an investigation of the correlation between the sutable conformation of a flexible ligand and the topology of the coordination network formed is a challenging task for inorganic chemists.

On the other hand, among the N-donor bridging ligands, bis(imidazole) ligands, as an important family of flexible N-donor ligands, have attracted great interest [14]. In our previous work [15], we have reported a series of fascinating archetypal structures based on the 1,4-bis(imidazol-1-ylmethyl)benzene (Bix) or 4,4'-bis(imidazol-1-ylmethyl)biphenyl (Bimb) ligands. On careful inspection of the reported cases, we found that the flexible nature of the alkyl ($-CH_2-$) spacer allows the ligands to bend and rotate freely so as to conform to the coordination geometries of central metal atoms. 1,3,5-Tris(imidazol-1-ylmethyl)-2,4,6-trimethylbenzene) (Titmb) is a flexible tri(imidazole) ligand that was concerned in recent years as a result of their pluridentated and excellent coordinated ability [16]. Herein, we report the syntheses and structural characterization of two coordination polymers, $[Zn_2(CAM)_2(Titmb)]$ (**I**) and $[Zn_4Co(OH)_2(BTEC)_2(Titmb)_2(H_2O)_2] \cdot 2H_2O$ (**II**), where H_2CAM = camphor acid, H_4BTEC = 1,2,4,5-benzenetetracarboxylic acid. Moreover, the luminescent properties of **I** have been investigated in the solid state at room temperature.

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[1] The article is published in the original.

EXPERIMENTAL

All chemicals and solvents were of reagent grade and used as received without further purification. The Titmb ligand was synthesized according to the reported method [17]. Elemental analysis was carried out on a PerkinElmer 240C elemental analyzer. The content of Co and Zn ion is based on ICP analysis. IR spectra were recorded on a Bruker Vector 22 FT-IR spectrophotometer by using KBr discs. The luminescent spectra for the powdered solid samples were measured at room temperature on a Hitachi F-4500 fluorescence spectrophotometer with a xenon arc lamp as the light source. In the measurements of emission and excitation spectra the pass width is 5 nm. All the measurements were carried out under the same experimental conditions.

Synthesis of I. A mixture containing $Zn(NO_3)_2 \cdot 6H_2O$ (59.5 mg, 0.2 mmol), H_2CAM (20.0 mg, 0.1 mmol), Titmb (36.0 mg, 0.1 mmol), and $LiOH \cdot H_2O$ (8.4 mg, 0.2 mmol) in 15 mL deionized water was sealed in a 25 mL Teflon lined stainless steel container and heated at 140°C for 3 days. Colorless block crystals of **I** were collected by filtration and washed with water and ethanol several times with a yield of 65%.

IR (KBr; ν, cm^{-1}): 3467 br, 3052 m, 2957 w, 2813 w 1609 m, 1572 s, 1549 m, 1520 m, 1441 m, 1387 s, 1321 w, 1283 w, 1137 w, 1091 w, 1029 w, 951 m, 826 w, 763 w, 721 w, 651 w, 545 w.

For $C_{41}H_{52}N_6O_8Zn_2$

anal. calcd., %:	C, 55.48;	H, 5.90;	N, 9.47.
Found, %:	C, 55.45;	H, 5.92;	N, 9.44.

Synthesis of II. A mixture of $Zn(NO_3)_2 \cdot 6H_2O$ (29.7 mg, 0.1 mmol), $Co(NO_3)_2 \cdot 6H_2O$ (29.1 mg, 0.1 mmol), H_4BTEC (25.4 mg, 0.1 mmol), Titmb (36.0 mg, 0.1 mmol), NaOH (16 mg, 0.4 mmol), and H_2O (15 mL) was added in a Teflon-lined stainless steel vessel. The vessel was sealed and heated for 3 days at 160°C. After the mixture was slowly cooled to room temperature, pale purple needle crystals of **II** were obtained in the yield of 52% Co.

IR (KBr, ν, cm^{-1}): 3367 br, 3125 m, 1608 s, 1577 s, 1545 s, 1519 m, 1437 m, 1389 s, 1351m, 1319 m, 1294 m, 1119 m, 1107 m, 1091 s, 855 w, 764 m, 735 s, 708 w, 671 w, 579 w.

For $C_{62}H_{62}N_{12}O_{22}CoZn_4$

anal. calcd., %:	C, 45.19;	H, 3.79;	N, 10.20;	Zn, 15.87;	Co, 3.58.
Found, %:	C, 45.22;	H, 3.91;	N, 10.12;	Zn, 15.84;	Co, 3.55.

X-ray diffraction analysis. Single-crystal data for complexes **I** and **II** were obtained on a Bruker Smart Apex II CCD with graphite-monochromated MoK_α radiation ($\lambda = 0.71073$ Å) at 291(2) K using the ω scan technique. The data were integrated by using the SAINT program [18], which was also used for the intensity corrections for the Lorentz and polarization effects. An empirical absorption correction was applied using the SADABS program [19]. The structures were solved by direct methods using the program SHELXS-97 [20], and all non-hydrogen atoms were refined anisotropically on F^2 by the full-matrix least-squares technique using the SHELXL-97 crystallographic software package [21]. The hydrogen atoms were generated geometrically. All calculations were performed on a personal computer with the SHELTXL crystallographic software package [21]. The details of the crystal parameters, data collection, and refinements for **I** and **II** are summarized in Table 1. Selected bond lengths and bond angles with their estimated standard deviations are listed in Table 2. Atomic coordinates and other structural parameters have been deposited with the Cambridge Crystallographic Data Centre (nos. 899225 and 804354; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

The asymmetric unit of **I** contains Zn(1) and Zn(2) cations, both lying in general positions, two crystallographically independent CAM^{2-} dianions and one Titmb ligand. As shown in Fig. 1a, Zn(1) ion is coordinated by three carboxylate oxygen atoms from two different CAM^{2-} dianions and two nitrogen atoms from two individual Titmb ligands to form a distorted trigonal bipyramidal geometry. The Zn(2) ion is surrounded by three carboxylate oxygen atoms from two independent CAM^{2-} dianions and one nitrogen atom from the Titmb ligand to construct a distorted tetrahedral coordination geometry. The Zn–O distances range from 1.863(3) to 2.099(3) Å, and the Zn–N distance is the range of 1.931(4)–2.050(4) Å. The O(N)ZnO(N) bond angles range from 55.29(13)° to 143.69(13)°, which are similar to those observed in the reported complexes [22]. It is interesting to note that the two types of CAM^{2-} dianions show different coordination modes. One bridges two Zn atoms with both two carboxylate groups in bidentate chelating coordination modes, while the other connects three Zn atoms with one of two carboxylate groups in a bidentate chelating coordination mode and the other one in a bidentate bridging coordination mode. As such, CAM^{2-} dianions bridge Zn^{2+} ions to form a 1D $[Zn(CAM)]_n$ double chain with $[Zn_4(CAM)_4]$ loops (Fig. 2). Interestingly, these 1D $[Zn(CAM)]_n$ double-chains are further linked by titmb ligands with *cis*, *trans*, *trans* conformation to generate a 3D framework (Fig. 3).

Better insight into such elegant frameworks can be achieved using the topology method: first, based on the considerations of the their connectivity, Zn(1), the dimer Zn_2 and Titmb ligand are viewed to be four-,

Table 1. Crystallographic parameters and a summary of data collection and refinement for **I** and **II**

Parameter	Value	
	I	**II**
M	887.63	1647.65
Crystal size, mm	0.28 × 0.22 × 0.20	0.26 × 0.08 × 0.06
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a, Å	11.7676(14)	11.7676(14)
b, Å	16.636(2)	16.636(2)
c, Å	19.631(2)	19.631(2)
α, deg	90.441(2)	3843.0(8)
β, deg	98.357(3)	71.830(8)
γ, deg	100.260(2)	76.515(8)
V, Å^3	2413.6(5)	71.439(8)
Z	2	1
ρ_{calcd}, mg m^{-3}	1.221	1.649
$F(000)$	928	841
θ Range, deg	1.62–26.00	1.48–25.10
Reflections collected	13248	8127
Independent reflections (R_{int})	9278 (0.0212)	5727 (0.0402)
Number of parameters	523	466
Goodness-of-fit on F^2	1.033	0.973
Final R indices ($I > 2\sigma(I)$)	$R_1 = 0.0591$, $wR_2 = 0.1459$	$R_1 = 0.0531$, $wR_2 = 0.11096$
R indices (all data)	$R_1 = 0.0855$, $wR_2 = 0.1534$	$R_1 = 0.0965$, $wR_2 = 0.1264$
Large diff. peak and hole, e Å^{-3}	0.932/–0.603	0.900/–0.466

$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. $wR_2 = |\Sigma w(|F_o|^2 - |F_c|^2)|/\Sigma|w(F_o)^2|^{1/2}$, $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$. $P = (F_o^2 + 2F_c^2)/3$.

six-, and three-connected nodes; second, Zn–O/N coordination bonds, CAM ligands are simplified to be linear connectors; then the combination of nodes and connectors suggests the (3,4,6)-connected frameworks for the present case. The topological notation for such (3,4,6)-connected frameworks is $(4.6^4.8)_2(4^2.6^4.8^9)(6^2.8)_2$ net (Fig. 4). In the literatures [23–25], the (3,4)-, (3,6)- and (4,6)-connected frameworks are often encountered, but we are not aware of a precedent characterized by such topology like that found in **I**.

Single-crystal X-ray diffraction analysis revealed that **II** exhibits an interesting heterometallic 3D metal-organic framework. crystallizes in the triclinic with space group $P\bar{1}$. As shown in Fig. 1b, there are two crystallographically unique Zn atoms, half of a Co atom (the Zn : Co ratio of 4 : 1 based on ICP analysis), one BTEC anion, one hydroxyl, one Titmb ligand, one coordinated water molecule, and one solvated water molecule in the asymmetric unit of complex **II**. Both of two unique Zn atoms adopt a distorted tetrahedral geometry and coordinates to three oxygen at-

Table 2. Selected bond lengths and angles in **I** and **II***

Bond	*d*, Å	Bond	*d*, Å
	I		
Zn(1)–N(2)	2.050(4)	Zn(2)–O(7)	2.093(3)
Zn(1)–O(1)	2.088(3)	Zn(2)–O(8)	2.096(3)
Zn(1)–O(5)	2.099(3)	Zn(2)–O(4)$^{\#3}$	1.860(3)
Zn(1)–O(6)	2.099(3)	Zn(2)–N(6)$^{\#2}$	1.931(4)
Zn(1)–N(4)$^{\#1}$	2.001(4)		
	II		
Zn(1)–O(9)	1.927(4)	Zn(2)–N(5)$^{\#6}$	2.021(5)
Zn(1)–O(4)$^{\#4}$	1.971(4)	Co(1)–O(6)	2.031(4)
Zn(1)–O(2)	1.994(4)	Co(1)–O(6)$^{\#7}$	2.031(4)
Zn(1)–N(3)	2.009(5)	Co(1)–N(1)$^{\#5}$	2.129(5)
Zn(2)–O(9)	1.934(4)	Co(1)–N(1)$^{\#8}$	2.129(5)
Zn(2)–O(8)$^{\#5}$	1.965(4)	Co(1)–O(1*w*)	2.162(4)
Zn(2)–O(1)	2.006(4)	Co(1)–O(1*w*)$^{\#7}$	2.162(4)
Angle	ω, deg	Angle	ω, deg
	I		
N(4)$^{\#1}$Zn(1)N(2)	110.82(16)	O(1)Zn(1)O(6)	143.69(13)
N(4)$^{\#1}$Zn(1)O(1)	107.83(15)	O(5)Zn(1)O(6)	55.29(13)
N(2)Zn(1)O(1)	115.91(16)	O(4)$^{\#3}$Zn(2)N(6)$^{\#2}$	117.75(16)
N(4)$^{\#1}$Zn(1)O(5)	137.00(15)	O(4)$^{\#3}$Zn(2)O(7)	108.42(14)
N(2)Zn(1)O(5)	92.69(14)	N(6)$^{\#2}$Zn(2)O(7)	132.64(15)
O(1)Zn(1)O(5)	91.95(13)	O(4)$^{\#3}$Zn(2)O(8)	120.77(15)
N(4)$^{\#1}$Zn(1)O(6)	90.81(14)	N(6)$^{\#2}$Zn(2)O(8)	101.21(14)
N(2)Zn(1)O(6)	83.95(14)	O(7)Zn(2)O(8)	60.64(13)
	II		
O(9)Zn(1)O(4)$^{\#4}$	124.65(16)	O(8)$^{\#5}$Zn(2)N(5)$^{\#6}$	100.32(19)
O(9)Zn(1)O(2)	113.21(17)	O(1)Zn(2)N(5)$^{\#6}$	109.31(19)
O(4)$^{\#4}$Zn(1)O(2)	114.55(15)	O(6)Co(1)O(6)$^{\#7}$	179.998(1)
O(9)Zn(1)N(3)	102.53(19)	O(6)Co(1)N(1)$^{\#5}$	89.88(17)
O(4)$^{\#4}$Zn(1)N(3)	98.91(19)	O(6)Co(1)N(1)$^{\#8}$	90.12(17)
O(2)Zn(1)N(3)	95.78(19)	O(6)Co(1)O(1*w*)	89.31(15)
O(9)Zn(2)O(8)$^{\#5}$	142.52(17)	N(1)$^{\#5}$Co(1)O(1*w*)	93.4(2)
O(9)Zn(2)O(1)	103.26(17)	O(6)Co(1)O(1*w*)$^{\#7}$	90.69(15)
O(8)$^{\#5}$Zn(2)O(1)	94.84(16)	N(1)$^{\#5}$Co(1)O(1*w*)$^{\#7}$	86.6(2)
O(9)Zn(2)N(5)$^{\#6}$	104.08(19)		

* Symmetry codes: $^{\#1}x - 1, y, z$; $^{\#2}-x + 1, -y + 2, -z + 1$; $^{\#3}x + 1, y, z + 1$; $^{\#4}-x + 1, -y + 2, -z + 1$; $^{\#5}-x + 2, -y + 1, -z + 1$; $^{\#6}x, y + 1, z$; $^{\#7}-x + 2, -y + 1, -z$; $^{\#8}x, y, z - 1$.

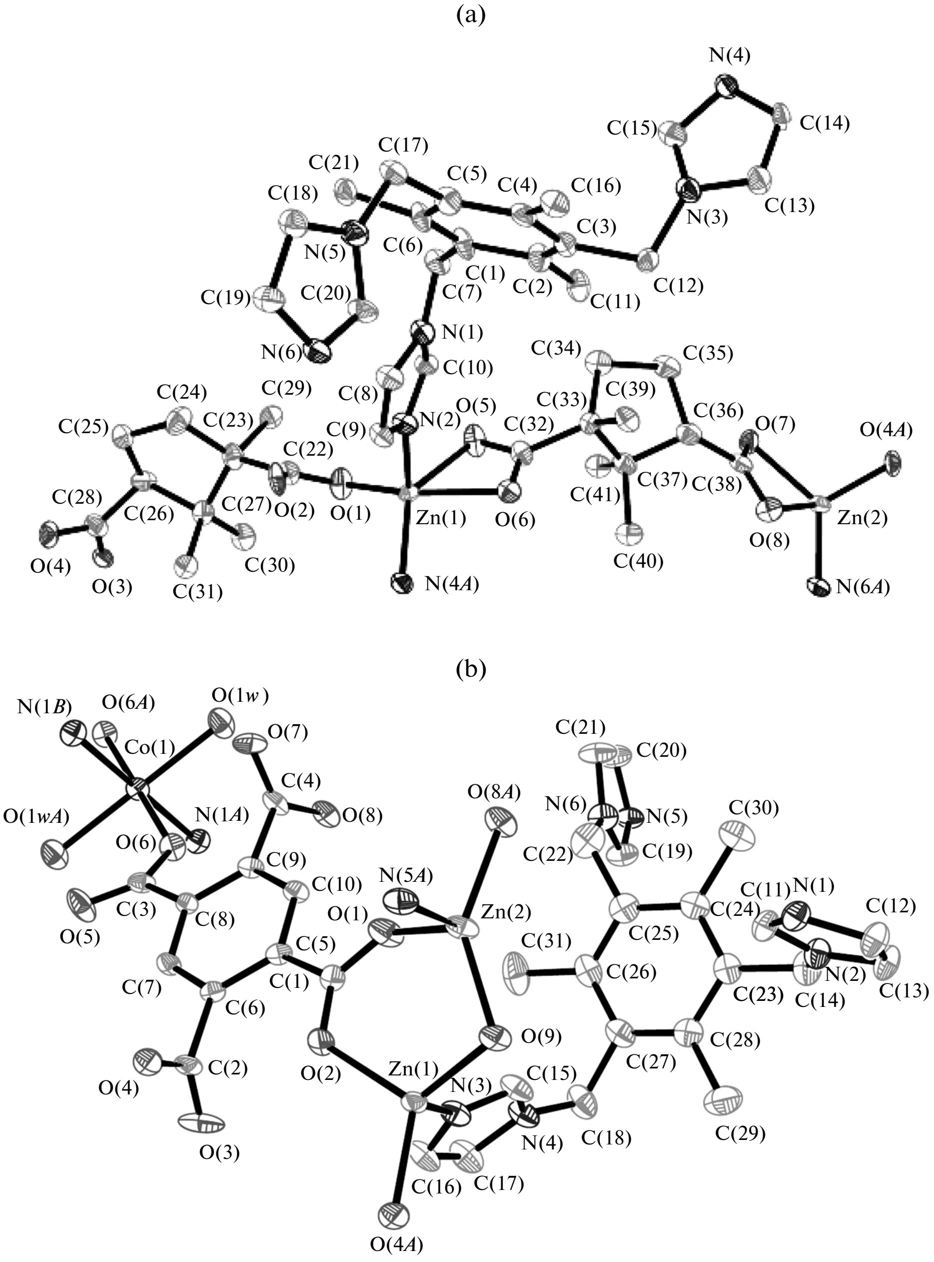

Fig. 1. Coordination environments of the Zn(II) atoms in **I** (a) and the Zn^{2+} and Co^{2+} ions in **II** (b) with the ellipsoids drawn at the 30% probability level (hydrogen atoms were omitted for clarity).

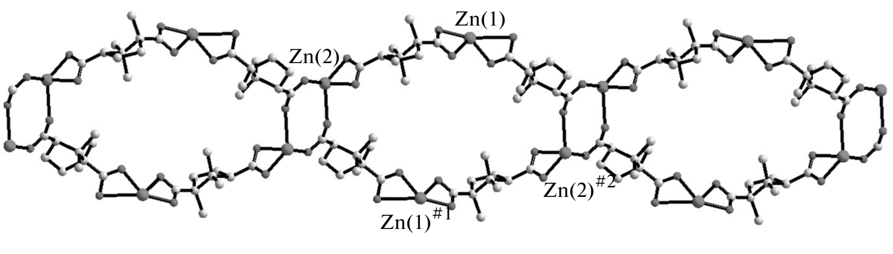

Fig. 2. The 1D double-chain structure with rhombic voids in complex **I**.

oms of two different carboxylate groups and one hydroxyl group with Zn–O distances in the range 1.927(4)–2.006(4) Å, and to one nitrogen atom of Titmb with Zn–N distances of 2.009(4) and 2.021(5) Å. The O(N)ZnO(N) bond angles vary from 94.84(16)° to 142.52(17)°. Two Zn atoms are connected through one bridging carboxylate group in a *syn-syn* conformation and one hydroxyl group to form a Zn_2 dimer with Zn···Zn separation of 3.128(2) Å. The Zn_2 dimers are linked by Btec ligands to furnish one-dimensional $\{Zn_2(OH)(BTEC)\}$ chains (Fig. 5). The Ni atoms are bound with two carboxylate oxygen atoms from Btec ligands, acting as connecting cations intercalated between the one-dimensional chains and a two-dimensional layer structure is generated (Fig. 6). The Co atoms are coordinated in slightly distorted octahedral geometries with Co–O bond distances ranging from 2.002(4) to 2.162(4) Å, which are the closest to the Ni–O bond distances (ranging from 2.0676(16) to 2.1280(13) Å) or the Co–O bond distances (ranging from 2.031(4) to 2.169(3) Å) in previously reported metal Btec complexes [26, 27]. Each plane for the carboxylate groups is twisted out of the benzene ring plane and the angles between the benzene ring plane and the each carboxylate group plane are 9.38° (for O(1)C(1)O(2) plane), 84.17° (for O(3)C(2)O(4) plane), 20.35° (for O(5)C(3)O(6) plane), and 74.99° (for O(7)C(4)O(8) plane). Each BTEC ligand acts as a μ_5-bridge linking four Zn atoms and one Co atoms with monodentate and bridging-bidentate coordination modes. The layers are further interlinked into a 3D framework via the Titmb ligands (Fig. 7). Each Titmb ligand bridges two Zn atoms and one Co atom in *cis*, *trans*, *trans* conformation.

Luminescent complexes are currently of great interest because of their various applications in chemical sensors, photochemistry, and electroluminescent display [28, 29]. The luminescent properties of Zn and Cd carboxylate compounds have been investigated [30]. The photoluminescent spectra of **I** and free neutral Titmb ligand were measured at room temperature. The intense emission band at 459 nm ($\lambda_{ex} = 365$ nm) is observed for **I**. The free Titmb ligand exhibits fluorescent emission bands at 423 nm ($\lambda_{ex} = 330$ nm). In order to understand the nature of the emission band, the photoluminescence properties of the H_2CAM ligand were analyzed. A weak emission ($\lambda_{max} = 561$ nm) could be observed. In comparison to the free ligands, the emission maximums of **I** have changed. This may be caused by a change in the HOMO and LUMO energy levels of deprotonated CAM^{2-} dianions and neutral ligands coordinating to metal centers, a charge-transfer transition between ligands and metal centers, and a joint contribution of the intraligand transitions or charge-transfer transitions between the coordinated ligands and the metal centers [31, 32]. These observations indicate that complex **I** may be excellent candidates for potential photoactive materials.

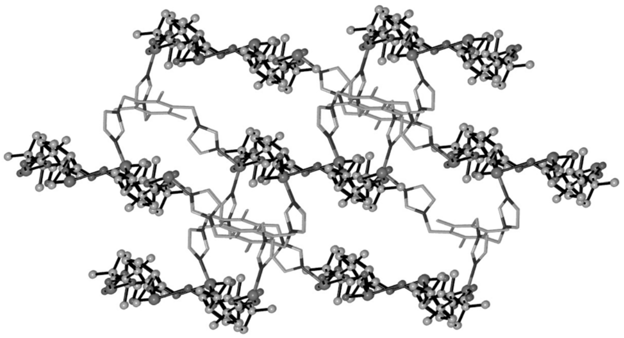

Fig. 3. View of a 3D framework extended by the Titmb ligands in **I**.

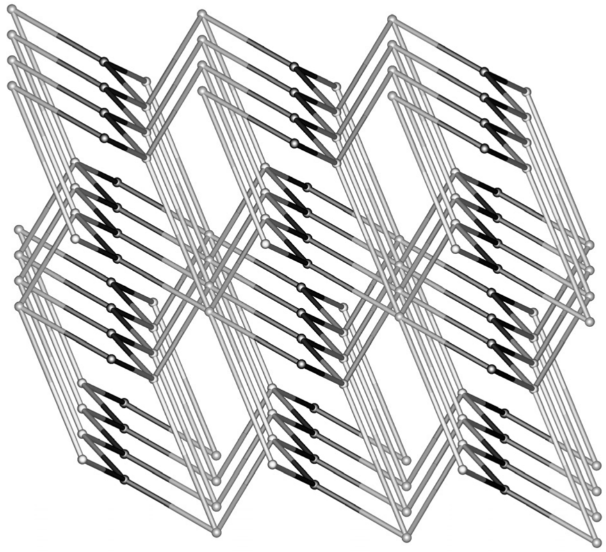

Fig. 4. Topological illustration for the trinodal (3,4,6)-connected $(4.6^4.8)_2(4^2.6^4.8^9)(6^2.8)_22$ net of **I**.

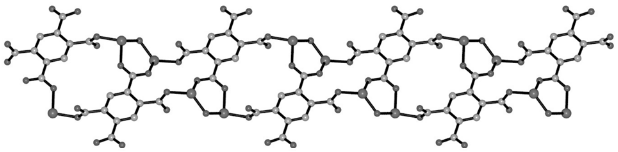

Fig. 5. View of one-dimensional $\{Zn_2(OH)(BTEC)\}$ chain in **II**.

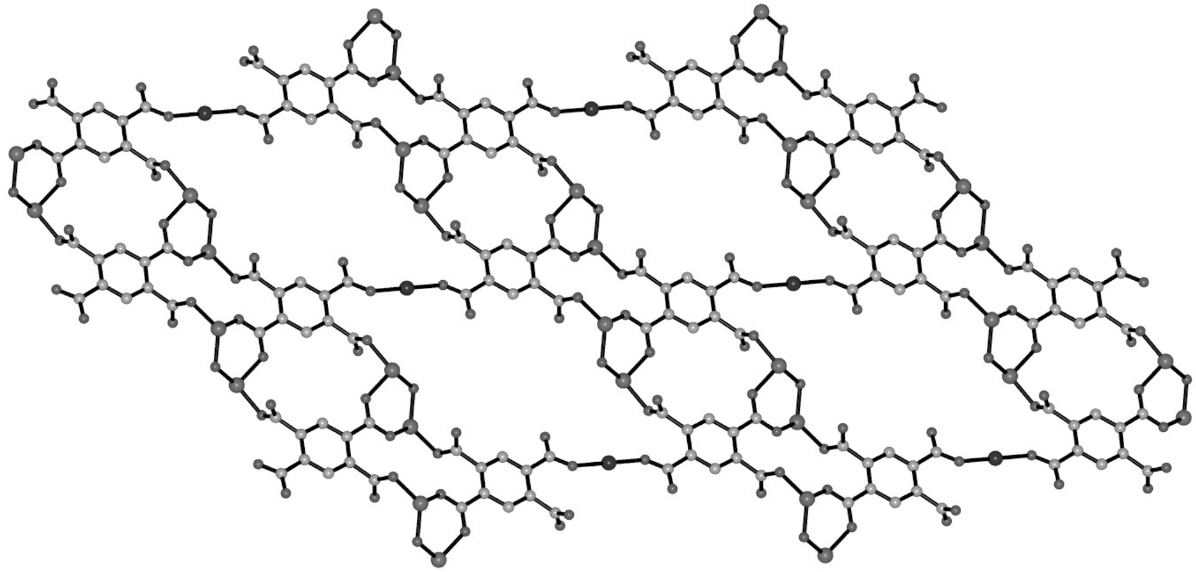

Fig. 6. View of two-dimensional layer structure in **II**.

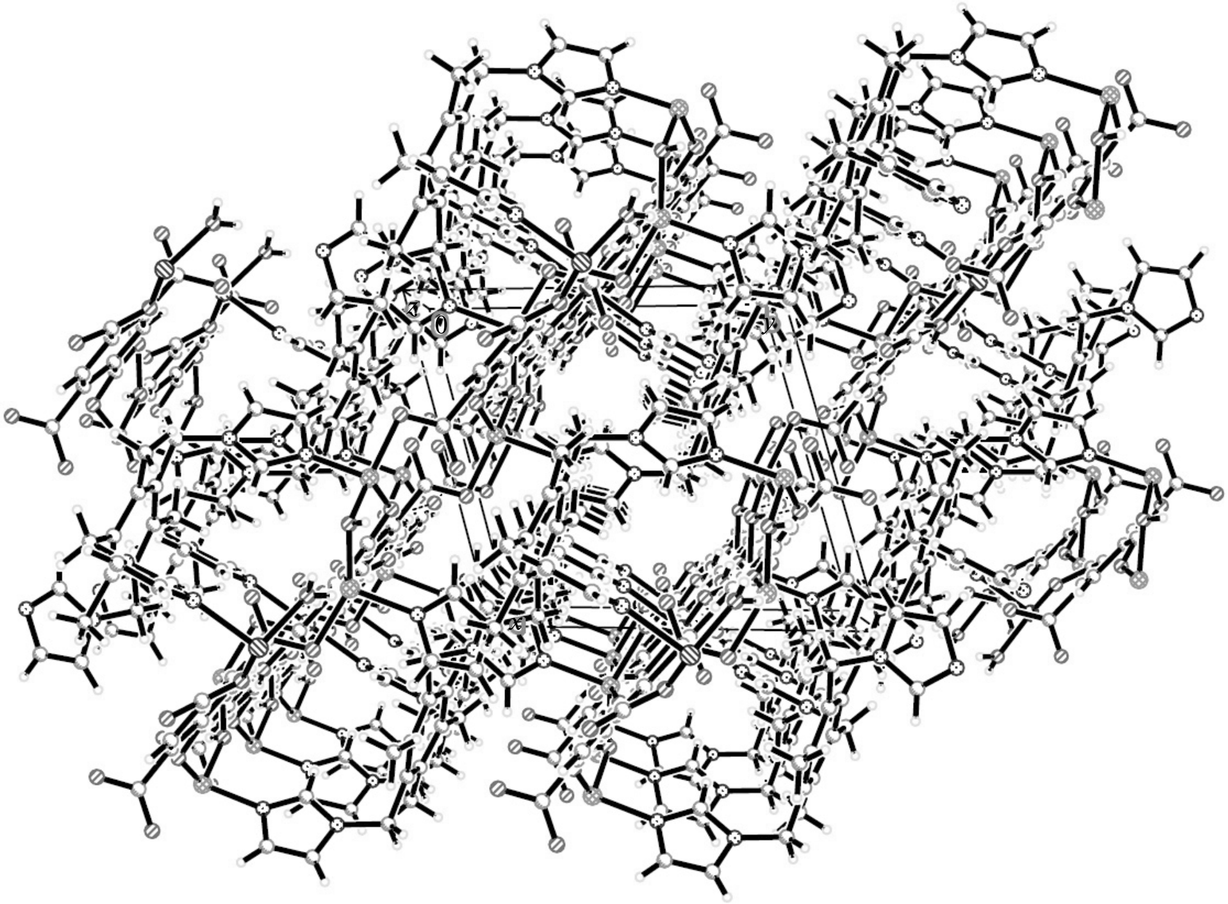

Fig. 7. The 3D framework constructed with the corresponding 2D layer by Titmb ligands.

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