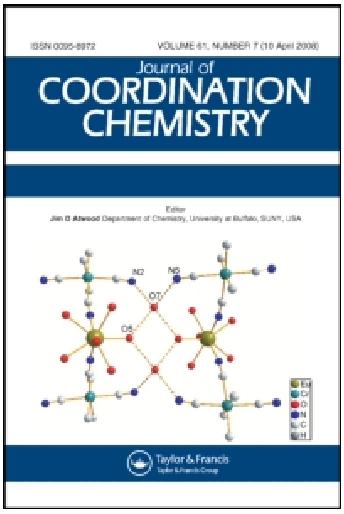

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Syntheses and crystal structures of copper(II) complexes derived from 2,4,6-tris(2-pyridyl)-1,3,5-triazine

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Treatment of freshly precipitated $Cu(OH)_2 \cdot xH_2O$ and 2,4,6-tris(2-pyridyl)-1,3,5-triazine (tptz) with oxalic and malonic acids in methanol-water at room temperature gave $[Cu(tptz)(C_2O_4)(H_2O)] \cdot 4H_2O$ (**1**) and $[Cu(pma)(C_3H_2O_4)(H_2O)] \cdot H_2O$ (**2**) (pma = 2-aminocarbonylpyridine), respectively. Reaction in the absence of any acid resulted in $[Cu(bpca)(tca)] \cdot 2H_2O$ (**3**) (bpca = bis(2-pyridylcarbonyl)amide anion; tca = 2-pyridinecarboxylate anion). Complex **1** consists of $[Cu(tptz)(C_2O_4)(H_2O)]$ and lattice H_2O molecules; the tridentate tptz ligand, bidentate oxalate dianion and an aqua ligand are bound to Cu with distorted octahedral geometry. Complex **2** is composed of $[Cu(pma)(C_3H_2O_4)(H_2O)]$ and lattice H_2O molecules; the bidentate 2-aminocarbonylpyridine ligand, a bidentate malonate dianion and an aqua ligand are coordinated to Cu with a slightly distorted square pyramidal geometry. Complex **3** consists of [Cu(bpca)(tca)] and lattice H_2O molecules. Square pyramidally coordinated Cu atoms are surrounded by tridentate bpca with nitrogen donor atoms and a bidentate 2-pyridinecarboxylate anion.

Keywords: Copper(II); N donors; 2,4,6-Tris(2-pyridyl)-1,3,5-triazineTptz); Crystal structures; Ligand reactions

1. Introduction

As an interesting polydentate nitrogen donor ligand, 2,4,6-tris(2-pyridyl)-1,3,5-triazine (tptz) has attracted increasing attention in the synthesis of novel transition metal complexes [1–6]. A literature survey shows two cases when tptz is used to prepare complexes. Tptz can remain intact in combination with metal ions [7–10], or undergoes decomposition upon coordination [11–15]. Recently, we have been interested in exploring the chemistry of transition metal complexes derived from tptz and dicarboxylic acids. Although a large number of mixed ligand complexes of dicarboxylic acids and phenanthroline have been prepared [16–21], related complexes of dicarboxylic acids and tptz have never been reported. Here we report the synthesis and structural

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characterization of three copper(II) complexes, $[Cu(tptz)(C_2O_4)(H_2O)] \cdot 4H_2O$ (**1**), $[Cu(pma)(C_3H_2O_4)(H_2O)] \cdot H_2O$ (**2**) (pma = 2-aminocarbonylpyridine) and [Cu(bpca)(tca)] $\cdot$ $2H_2O$ (**3**) (bpca = bis(2-pyridylcarbonyl)amide anion, tca = 2-pyridinecarboxylate anion). To the best of our knowledge, complex **1** is the first example of a copper(II) complex prepared in aqueous media with intact tptz. Complexes **2** and **3**, with hydrolyzed tptz, are unprecedented.

2. Experimental

2.1. *Physical measurements*

All chemicals of *p.a.* grade were commercially available and used without further purification. C, N and H microanalyses were performed with a Perkin Elmer 2400II CHNS/O instrument. FT-IR spectra were recorded using KBr pellets in the range 4000–400 cm^{-1} on a Shimadzu FTIR-8900 spectrophotometer.

2.2. *Syntheses*

Freshly precipitated $Cu(OH)_2 \cdot xH_2O$ was used for the syntheses of the complexes. Some 2.0 cm^3 of aqueous NaOH (1M) was added drop wise to an aqueous solution of $CuCl_2 \cdot 2H_2O$ (0.171 g, 1.00 mmol) in 1.0 cm^3 of H_2O to produce a blue precipitate, which was then centrifuged and washed with doubly distilled water several times until no Cl^- ions were detected in the supernatant.

2.2.1. $[Cu(tptz)(C_2O_4)(H_2O)] \cdot 4H_2O$ (1). Freshly precipitated $Cu(OH)_2 \cdot xH_2O$ (from 0.171 g of $CuCl_2 \cdot 2H_2O$) was added to a mixture of tptz (0.312 g, 1.00 mmol) and oxalic acid (0.901 g, 1.00 mmol) in methanol/water (50 cm^3, 1 : 1, v/v). The solution was stirred and filtered. The filtrate (pH = 4.78) was allowed to stand at room temperature and green crystals grew during two days. Yield: ca. 45% based on initial $CuCl_2 \cdot 2H_2O$. IR (cm^{-1}): 3396 (H_2O), 1645, 1523, 1419 cm^{-1} (tptz). Anal. Calcd for $C_{20}H_{22}N_6O_9Cu$ (%): C, 43.36; H, 3.97; N, 15.17. Found: C, 42.99; H, 3.20; N, 15.01.

2.2.2. $[Cu(pma)(C_3H_2O_4)(H_2O)] \cdot H_2O$ (2). Complex **2** was prepared in the same way as above except malonic acid (0.104 g, 1.00 mmol) was used instead of oxalic acid. Slow evaporation of the filtrate (pH = 6.28) for a week at room temperature afforded blue crystals. Yield: ca 60% based on initial $CuCl_2 \cdot 2H_2O$. IR (cm^{-1}): 3411 (H_2O), 1718 (C=O), 1631, 1450 (pma). Anal. Calcd for $C_9H_{12}N_2O_7Cu$ (%): C, 33.38; H, 3.70; N, 8.65. Found C, 32.95; H, 3.21; N, 8.47.

2.2.3. [Cu(bpca)(tca)] $\cdot$ $2H_2O$ (3). Fresh $Cu(OH)_2 \cdot xH_2O$ precipitate (from 0.171 g of $CuCl_2 \cdot 2H_2O$) was added to a solution of tptz (0.312 g, 1.00 mmol) in methanol/water (50 cm^3, 1 : 1, v/v). The resulting mixture was stirred and filtered. Blue crystals grew in the filtrate (pH = 11.54) by slow evaporation during two weeks at room temperature. Yield: ca 57% based on initial $CuCl_2 \cdot 2H_2O$. IR (cm^{-1}): 3400 (H_2O), 1700 (C=O), 1621, 1428 (bpca). Anal. Calcd for $C_{18}H_{18}N_4O_7Cu$ (%): C, 46.40; H, 3.86; N, 12.03. Found C, 46.15; H, 2.98; N, 11.93.

2.3. *Crystallography*

Suitable single crystals of **1**, **2**, and **3** were selected under a polarizing microscope and mounted on a Bruker P4 diffractometer with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) for cell determination and subsequent data collection. Lattice parameters were refined from 2θ values (10–25°) of 25 carefully centred reflections and reflection intensities with $2\theta_{max} = 55°$ were collected at 293 K using the θ–2θ scan technique. Data were corrected for L_P and absorption effects. The structures were solved by direct methods using the SHELXS-97 program [22]. Subsequent difference Fourier syntheses enabled all non-hydrogen atoms to be located. After several cycles of refinement, hydrogen atoms associated with C atoms were added in calculated positions and all remaining hydrogen atoms were derived from difference Fourier syntheses. Final full-matrix least-squares refinement using the SHELXL-97 program [23] converged well using anisotrpic thermal parameters for non-hydrogen atoms and isotropic thermal parameters for hydrogen atoms.

3. Results and discussion

3.1. *Synthesis and IR spectroscopy*

Reaction of tptz, oxalic acid and freshly precipitated $Cu(OH)_2 \cdot xH_2O$ in methanol-water at room temperature afforded $[Cu(tptz)(C_2O_4)(H_2O)] \cdot 4H_2O$ (**1**). Substitution of malonic acid for oxalic acid yielded $[Cu(pma)(C_3H_2O_4)(H_2O)] \cdot H_2O$ (**2**), indicating the starting tptz ligand to be hydrolyzed to 2-aminocarbonylpyridine (pma). In the absence of any acid, reaction of tptz and freshly precipitated $Cu_2(OH)_3Cl$ produced $[Cu(bpca)(tca)] \cdot 2H_2O$ (**3**), suggesting that under the strongly basic conditions tptz is hydrolyzed to form bis(2-pyridylcarbonyl)amide (bpca) and 2-pyridinecarboxylate (tca). In IR spectra of **1–3**, broad bands of moderate intensity due to $\nu(H_2O)$ appear at 3396, 3411, and 3400 cm^{-1}, respectively. Intense, sharp multiple peaks assignable to ν(pyridyl) fall in the range 1400 to 1630 cm^{-1}. Strong peaks attributed to the carbonyl group appear at 1645 (**1**), 1718 (**2**), and 1700 cm^{-1} (**3**).

3.2. *Structures of the complexes*

Figure 1 shows ORTEP drawings of **1**, **2** and **3** with corresponding atom numbering schemes. Crystal data are listed in table 1 and selected bond distances and angles are given in table 2. Compound **1** consists of lattice water and $[Cu(tptz)(C_2O_4)(H_2O)]$ molecules, in which the Cu atoms are in a distorted octahedral coordination environment defined by three N atoms of one tridentate tptz ligand and three O atoms of one aqua ligand and an oxalate ion. The plane comprising N(1), N(4), and N(3) is orientated nearly perpendicular to that defined by O(1), O(3), and O(5). Cu–N distances vary from 2.038 to 2.416 Å, and the Cu–O bond distances fall in the region

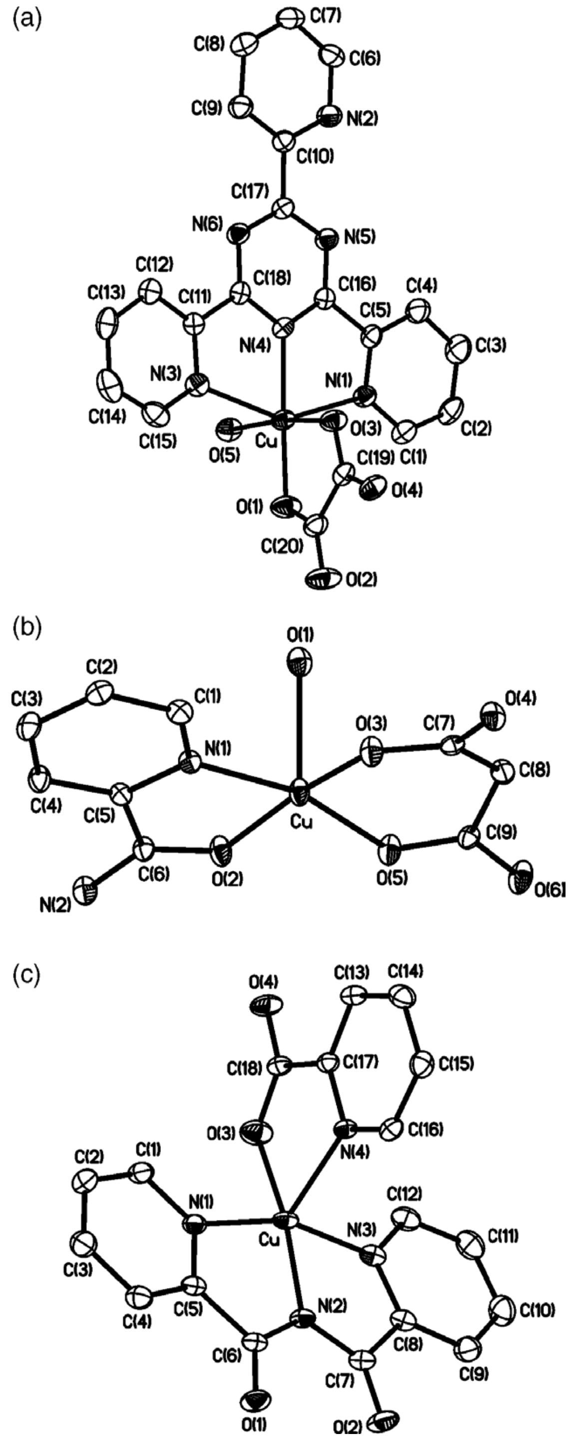

Figure 1. ORTEP view of (a) [Cu(tptz)(C_2O_4)(H_2O)], (b) [Cu(pma)($C_3H_2O_4$)(H_2O)] and (c) [Cu(bpca)(tca)] in **1**, **2** and **3**, respectively, with corresponding atom labelling. Displacement ellipsoids are drawn at the 45% probability level.

Table 1. Crystal data and refinement details for **1**, **2** and **3**.

	1	**2**	**3**
Empirical formula	$C_{20}H_{22}CuN_6O_9$	$C_9H_{12}CuN_2O_7$	$C_{18}H_{16}CuN_4O_6$
Formula mass	553.98	323.75	447.89
Space group ($P\bar{1}$)			
a [Å]	7.797(2)	8.069(2)	8.371(1)
b [Å]	11.881(2)	8.770(2)	10.751(1)
c [Å]	14.051(3)	10.429(2)	11.861(1)
α[°]	97.43(1)	68.75(2)	108.993(8)
β [°]	102.23(2)	81.38(2)	106.645(9)
γ [°]	108.41(2)	63.42(2)	99.191(9)
V [Å^3]	1179.6(4)	615.0(3)	928.6(2)
Z	2	2	28
ρ (calcd) [Mg m^{-3}]	1.560	1.748	1.602
μ [m^{-1}]	0.989	1.807	1.221
F(000)	570	330	458
Crystal size [mm^3]	0.56 × 0.22 × 0.05	0.24 × 0.11 × 0.07	0.38 × 0.29 × 0.13
θ range [°]	1.52–27.50	2.10–27.50	1.95–27.49
hkl ranges	−9/1, −14/15, −18/18	−10/1, −10/9, −13/13	−10/1, −12/12, −15/15
Reflections collected	9143	3356	4990
Independent reflections	5375	2768	4139
Observed reflections	3761	2187	3552
R_{int}	0.0385	0.0431	0.0207
Goodness-of-fit on F^2	0.990	1.089	1.050
R_1^a	0.0485	0.0434	0.0341
wR_2^b	0.1072	0.0979	0.0861

[a] $R = \Sigma\|F_o|-|F_c\|/\Sigma F_o$; [b] $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

Table 2. Selected bond distances (Å) and angles (°) for **1**, **2** and **3**.

1		**2**		**3**	
Cu–O1	1.929(2)	Cu–O1	2.334(3)	Cu–O3	1.951(2)
Cu–O3	1.993(2)	Cu–O2	1.960(2)	Cu–N1	2.046(2)
Cu–O5	2.018(2)	Cu–O3	1.921(2)	Cu–N2	1.952(2)
Cu–N1	2.330(3)	Cu–O5	1.918(2)	Cu–N3	2.047(2)
Cu–N3	2.416(3)	Cu–N1	2.004(3)	Cu–N4	2.242(2)
Cu–N4	2.038(2)				
O1–Cu–O3	83.84(9)	O1–Cu–O2	97.0(1)	O3–Cu–N1	97.80(9)
O1–Cu–O5	89.4(1)	O1–Cu–O3	93.3(1)	O3–Cu–N2	169.25(8)
O1–Cu–N1	104.8(1)	O1–Cu–O5	100.6(1)	O3–Cu–N3	98.47(9)
O1–Cu–N3	107.0(1)	O1–Cu–N1	90.4(1)	O3–Cu–N4	78.80(7)
O1–Cu–N4	177.78(9)	O2–Cu–O3	169.2(1)	N1–Cu–N2	80.94(8)
O3–Cu–O5	172.71(9)	O2–Cu–O5	86.8(1)	N1–Cu–N3	159.92(7)
O3–Cu–N1	94.01(9)	O2–Cu–N1	81.1(1)	N1–Cu–N4	96.39(8)
O3–Cu–N3	90.29(9)	O3–Cu–O5	94.5(1)	N2–Cu–N3	80.87(8)
O3–Cu–N4	94.00(9)	O3–Cu–N1	95.6(1)	N2–Cu–N4	111.94(7)
O5–Cu–N1	90.2(1)	O5–Cu–N1	164.6(1)	N3–Cu–N4	98.24(8)
O5–Cu–N3	89.2(1)				
O5–Cu–N4	92.8(1)				
N1–Cu–N3	148.23(8)				
N1–Cu–N4	74.79(9)				
N3–Cu–N4	73.51(9)				

1.929 to 2.018 Å (table 2). The Cu–N distances to N(1) and N(3) are longer due to the Jahn–Teller effect. The N(1)–Cu–N(3) bond angle of 148.23° exhibits considerable deviation from the ideal value of 180° due to the geometric impositions of the five-membered rings. Lattice water molecules are hydrogen bonded to one another to form 1D chains consisting alternatively of rhombic and chair-shaped hexagonal rings (figure 2). Along [100] [Cu(Tptz)(C_2O_4)(H_2O)] molecules are linked to neighbours through hydrogen bonds between aqua and non-coordinated oxalate oxygen atoms ($d(O(5)–H \cdots O(4)^I) = 2.752$ Å, $\angle(O(5)–H \cdots O(4)^I) = 168$, I: $x+1$, y, z) into infinite chains. Tptz ligands of one chain protrude into grooves between adjacent tptz ligands of a neighbouring chain, the arrangement being stabilized by weak C–H$\cdots$O and $\pi \cdots \pi$ stacking interactions between aromatic rings. These in turn are assembled via $\pi \cdots \pi$ stacking interactions into 2D layers parallel to (010) as illustrated in figure 2. They are stacked along [010] with the hydrogen bonded water molecule chains

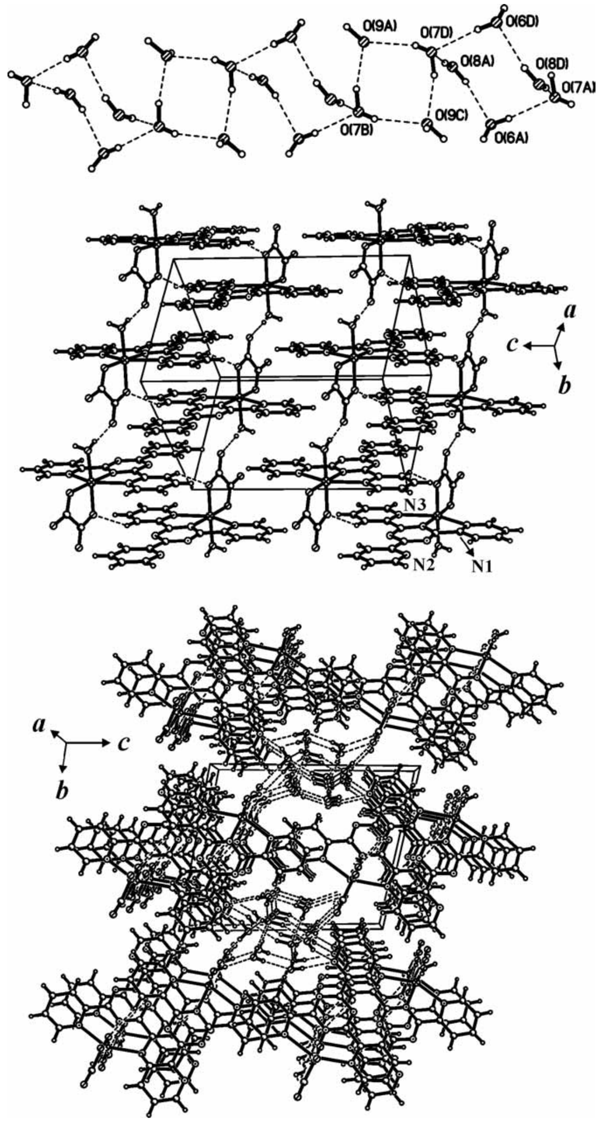

Figure 2. Hydrogen bonded chains of water molecules in **1** (top); [Cu(tptz)(C_2O_4)(H_2O)] molecules assembled into supramolecular layers (middle); crystal structure of **1** (bottom). Hydrogen bonds are indicated by dashed lines.

sandwiched between them (figure 2). It is clear that the crystal structure is significantly stabilized by the extensive hydrogen bonding scheme.

Compound **2** is built up of the [Cu(pma)($C_3H_2O_4$)(H_2O)] and lattice H_2O molecules (figure 3). Within the complex molecule, the Cu atom is square pyramidally coordinated

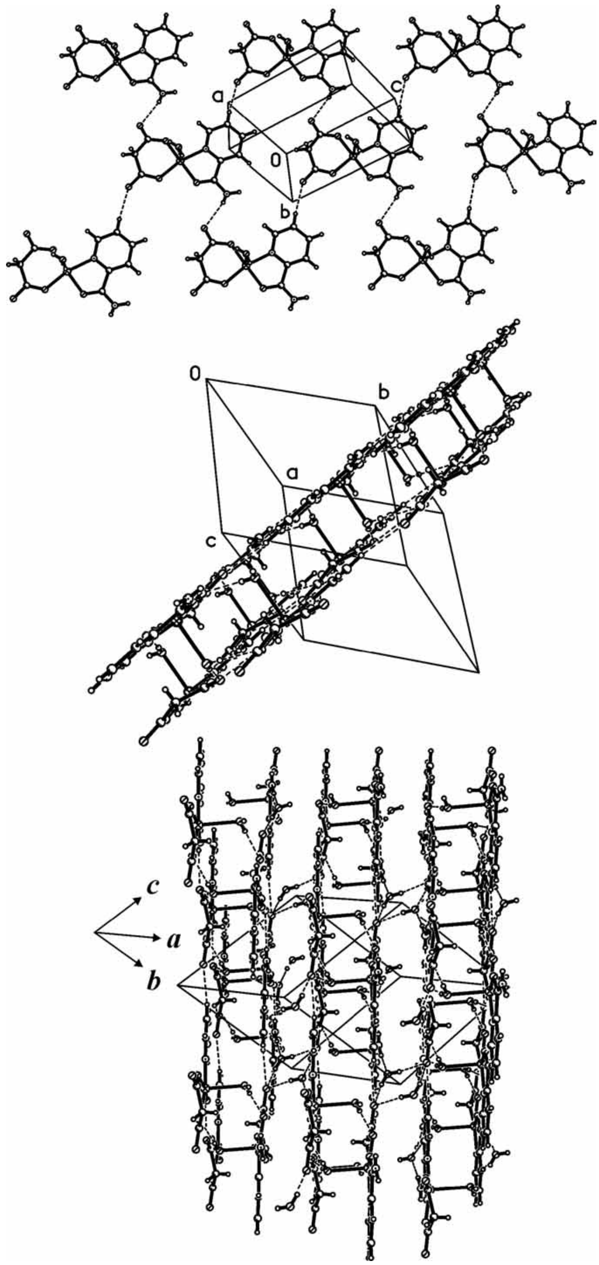

Figure 3. Supramolecular layer formed from [Cu(pma)($C_3H_2O_4$)(H_2O)] molecules by hydrogen bonds indicated by dashed lines (top); bi-layer generated by [Cu(pma)($C_3H_2O_4$)(H_2O)] molecules (middle); crystal structure of **2** (bottom). Hydrogen bonds are indicated by dashed lines.

by one nitrogen and four oxygen atoms. The aqua ligand is situated at the apical position and the basal plane of the coordination geometry is defined by carboxide oxygen and imine nitrogen atoms of 2-aminocarbonylpyridine and two carboxylate oxygen atoms of a bidentate, chelating malonate ion. The apical Cu–O bond distance is 2.334 Å and equatorial bond lengths fall in the range 1.918 to 1.960 Å, with Cu–N = 2.004 Å (table 2). The Cu atom is shifted by 0.18 Å from the basal plane toward the apical aqua ligand. The amine group of one [Cu(pma)($C_3H_2O_4$)(H_2O)] molecule forms an intermolecular hydrogen bond to the uncoordinated malonate oxygen atom

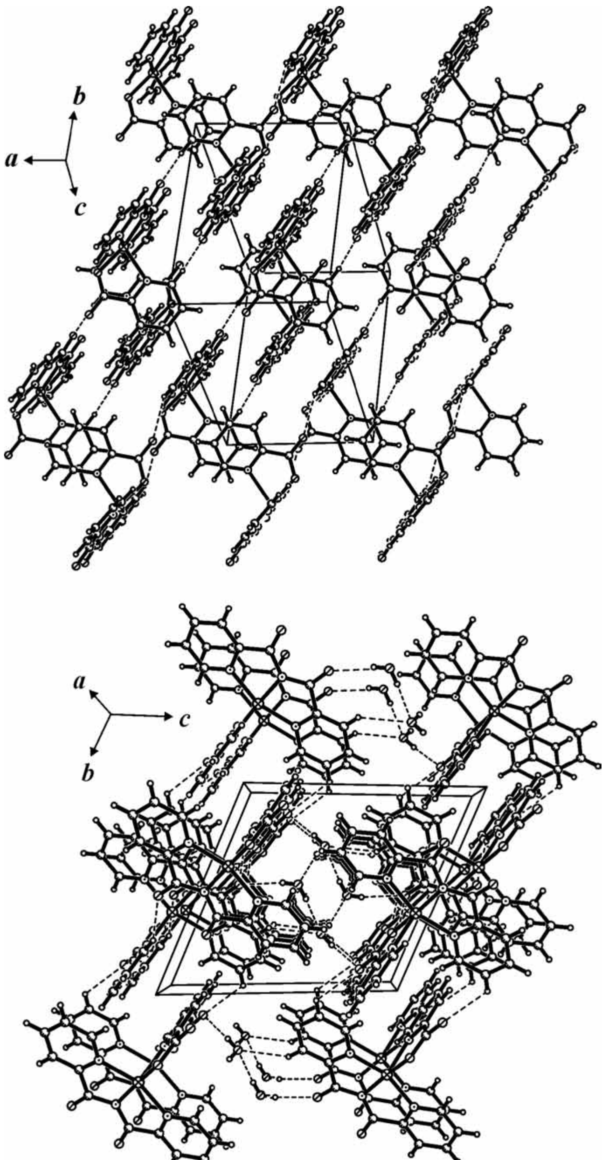

Figure 4. Supramolecular assembly of [Cu(bpca)(tca)] molecules (top); crystal structure of **3** (bottom). Hydrogen bonds are indicated by dashed lines.

of a neighboring complex, resulting in 2D layers (d(N2–H $\cdots$ $O4^{I}$) = 2.962 Å, I: $x-1$, $y+1$, z). Adjacent layers are held together by interlayer hydrogen bonds from aqua ligands to both coordinated O(5) and uncoordinated (O4) malonate oxygen atoms (d(O1–H $\cdots$ $O5^{II}$) = 2.910 Å, d(O1–H $\cdots$ $O4^{III}$) = 2.856 Å, II: $-x+2$, $-y+2$, $-z+1$, III: $-x+3$, $-y+1$, $-z+1$) to form bi-layers with *endo*-oriented aqua ligands (figure 3). Lattice water molecules are located between the bi-layers and function as connectors through hydrogen bonds to uncoordinated malonate O(6) atoms and amine groups (d(O7–H $\cdots$ $O6^{IV}$) = 2.957 Å, d(O7–H $\cdots$ $O6^{V}$) = 2.742 Å, d(N2–H $\cdots$ O7) = 2.840 Å, IV: $-x+1$, $-y+2$, $-z+1$, V: $x-1$, y, $z+1$).

In complex **3**, bis(2-pyridylcarbonyl)amide (bpca) and 2-pyridinecarboxylate (tca) anions formed by hydrolysis of tptz serve as tridentate N-donor and bidentate N,O-donor ligands, respectively. They coordinate the Cu atoms to form [Cu(bpca)(tca)] with distorted square pyramidal geometry. The basal plane is defined by one tca oxygen atom (O3) and three bpca nitrogen atoms (N1, N2, N3); the tca imine nitrogen atom (N4) occupies the apical position (d(Cu–N4) = 2.242(2), d(Cu–O3) = 1.951(2), basal d(Cu–N) = 1.952 to 2.047 Å, see table 2). Cu atoms are displaced by 0.18 Å from the basal plane toward the apical imine N atom. Weak intermolecular C–H $\cdots$ O hydrogen bonds between neighbouring [Cu(bpca)(tca)] molecules (d(C16–H $\cdots$ $O1^{I}$) = 3.250 Å, I: $-x+2$, $-y+1$, $-z+2$; d(C2–H $\cdots$ $O4^{II}$) = 3.147 Å, II: $-x+1$, $-y+2$, $-z+2$) result in 2D layers parallel to (001) as shown in figure 4. The layers are stabilized by intermolecular $\pi \cdots \pi$ stacking interactions between bpca ligands (mean inter-planar distance 3.45 Å) and tca ligands (mean inter-planar distance 3.30 Å). The crystallographically independent lattice water molecules (O5, O6), which are sandwiched between the 2D layers (figure 4), are hydrogen bonded to one another to generate rhombic $(H_2O)_4$ clusters with d(O5–H $\cdots$ $O6^{III}$) = 2.823 Å (III: $x+1$, y, $z+1$) and d(O6–H $\cdots$ $O5^{IV}$) = 2.812 Å (IV: $-x+1$, $-y+1$, $-z+2$). The carbonyl O1 oxygen atoms simultaneously act as hydrogen bond acceptors from the lattice O5 water molecules (d(O5–H $\cdots$ O1) = 2.908 Å). In addition, the lattice O6 water molecules form hydrogen bonds to the uncoordinated O4 carboxylate atoms of tca anions with d(O6–H $\cdots$ O4) = 2.770 Å. According to the above description, it is clear that the extensive hydrogen bonding is responsible for assembly of [Cu(bpca)(tca)] and lattice water molecules to complete the crystal structure.

Supplementary data

Files CCDC–278635 (**1**), CCDC–278636 (**2**), CCDC–278637 (**3**) contain supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) +44 1223 336 033; Email: deposit@ccdc.cam.ac.uk].

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References

[1] C.R. De Silva, J. Wang, M.D. Carducci, S.A. Rajapakshe, Z. Zheng. *Inorg. Chim. Acta*, **357**, 630 (2004).
[2] R.M. Berger, D.D. Ellis II. *Inorg. Chim. Acta*, **241**, 1 (1996).
[3] R.M. Berger, J.R. Holcombe. *Inorg. Chim. Acta*, **232**, 217 (1995).
[4] N. Gupta, N. Grover, G.A. Neyhart, P. Singh, H.H. Thorp. *Inorg. Chem*., **32**, 310 (1993).
[5] S. Chirayil, V. Hegde, Y. Jahng, R.P. Thummel. *Inorg. Chem*., **30**, 2821 (1991).
[6] N.C. Thomas, B.L. Foley, A.L. Rheingold. *Inorg. Chem*., **27**, 3426 (1988).
[7] T. Glaser, T. Lügger, R. Fröhlich. *Eur. J. Inorg. Chem*., 394 (2004).
[8] S.A. Cotton, V. Franckevicius, J. Fawcett. *Polyhedron*, **21**, 2055 (2002).
[9] J. Granifo. *Polyhedron*, **18**, 1061 (1999).
[10] J. Dahlén, S. Karlsson. *J. Chromatogr. A*, **848**, 491 (1999).
[11] P. Paul. *Proc. Ind. Acad. Sci. (Chem. Sci.)*, **114**, 269 (2002).
[12] P. Paul, B. Tyagi, A.K. Bilakhiya, M.M. Bhadbhade, E. Suresh. *J. Chem. Soc., Dalton Trans*., 2009 (1999).
[13] A. Cantarero, J.M. Amigo, J. Faus, M. Julve, T. Debaerdemaeker. *J. Chem. Soc., Dalton Trans*., 2033 (1998).
[14] P. Paul, B. Tyagi, M.M. Bhadbhade, E. Suresh. *J. Chem. Soc., Dalton Trans*., 2273 (1997).
[15] J. Faus, M. Julve, J.M. Amigo, T. Debaerdemaeker. *J. Chem. Soc., Dalton Trans*., 1681 (1989).
[16] Y.Q. Zheng, J. Sun, J.L. Lin. *J. Mol. Struct*., **650**, 49 (2003).
[17] Y.Q. Zheng, J.L. Lin, B.Y. Chan. *J. Mol. Struct*., **646**, 151 (2003).
[18] Y.Q. Zheng, J.L. Lin, Z.P. Kong. *Z. Anorg. Allg. Chem*., **629**, 357 (2003).
[19] Y.Q. Zheng, J.L. Lin, Z.P. Kong. *Polyhedron*, **22**, 2699 (2003).
[20] Y.Q. Zheng, Z.P. Kong. *Inorg. Chem. Comm*., **6**, 478 (2003).
[21] P. Segl'a, M. Jamnický, M. Koman, J. Šima, T. Glowiak. *Polyhedron*, **17**, 4525 (1998).
[22] G.M. Sheldrick. *SHELXL–97, Program for Crystal Structure Refinement*, University of Göttingen, Germany (1997).
[23] G.M. Sheldrick. *SHELXS–97, Program for Crystal Structure Solution*, University of Göttingen, Germany (1997).