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Self-assembled carboxylate complexes of zinc, nickel and copper

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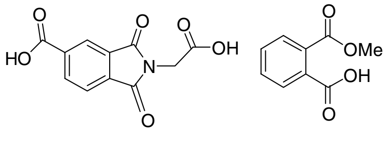
Abstract

A metallo-organic hybrid acid namely tetra-aquo bis-4-carboxy-*N*-phthaloylglycinato zinc(II) dihydrate is prepared and characterised. In this complex the hydrogen bonding by free carboxylic acid group and π–π interactions between the rings in crystal lattice contributes to the formation of self-assembled structure. A monomeric nickel complex from 2-carbomethoxy benzoic acid (**L2H**) and pyridine $[Ni(\mathbf{L2})(py)_3(H_2O)_2]\mathbf{L2}$ is prepared (where py = pyridine). This complex has ionic as well as monodentate carboxylates. It forms self-assembly by C–H···π as well as hydrogen-bonding interactions. The 2-carbomethoxy benzoic acid (**L2H**) forms dimeric copper complex $[Cu_2(\mathbf{L2})_4(H_2O)_2]2H_2O$ which has an extended chain structure through hydrogen-bond interactions.

Keywords: Metallo-organic hybrid acid; Self-assembly; Hydrogen-bonding; Carboxylate complexes; First row transition metal

1. Introduction

The metal carboxylate complexes are vastly studied [1–13]. The carboxylate ligand can have different binding modes and each of them may play role in the formation of supramolecular assembly [14–18]. The compounds having multiple carboxylic acid groups are good synthons for supramolecular architectures and co-ordination polymers [3–7]. Structural complexity arises in co-ordination polymers and metallo-organic hybrid systems due to variation of co-ordination number from metal to metal [19–23]. So, co-ordination behaviour of carboxylates in competitive environment needs to be understood at molecular level. We report here the co-ordination behaviour of 4-carboxy-*N*-phthaloylglycine (**L1H**) and 2-carbomethoxy benzoic acid (**L2H**) towards Zn(II), Ni(II) and Cu(II).



L1H **L2H**

2. Experimental

All the chemicals were obtained from Sigma–Aldrich Chemical Co. and used as obtained. The IR spectra were recorded on a Nicolet DSP 450 spectrometer and the UV–vis spectra were recorded in Hitachi U-3200 spectrophotometer. The NMR spectra were recorded in a Varian 400 MHz NMR machine and all the chemical shifts reported are in parts per million with reference to tetramethylsilane as internal standard. The X-ray diffraction data were collected on Bruker 3-circle SMART Apex diffractometer with CCD area detector, using graphite monochromated Mo–K_α radiation from a 60 W microfocus Bede Microsource® with glass polycapillary optics. The structures were solved by direct methods and refined by full-matrix least squares against F^2 of all data, using SHELXTL software

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by direct method. All the data collections were done at room temperature. The important crystallographic parameters of the reported complexes are given in Table 1.

2.1. Synthesis of metal complexes

2.1.1. Synthesis of 4-carboxy-N-phthaloylglycine (*L1H*)

A solution of 1,2,4-benzene tricarboxylic acid anhydride (1.92 g, 10 mmol) in glacial acetic acid (50 ml) at 50 °C was prepared in a round-bottomed flask. Glycine (0.750 g, 10 mmol) was added to this solution, the mixture refluxed for 6 h. The solution was then transferred to a beaker and concentrated to 25 ml by placing the beaker over a hot plate. On cooling and standing for overnight white microcrystalline precipitate appeared. The white precipitate was filtered and washed three times with distilled water to obtain the pure product. Yield: 86%. IR (KBr, cm^{-1}): 3066 (b), 2992(w), 2952(w), 2878(br), 2662(w), 2574(w), 1787(s), 1724(br s), 1600(w), 1416(s), 1398(s), 1296(s), 1262(s),1200(s), 970(w), 746(w), 726(w). ^1H NMR (DMSO-d_6): 13.5 (b, 2H), 8.4 (d, $J = 9.2$ Hz, 1H), 8.2 (s, 1H), 8.0(d, $J = 7.6$ Hz, 1H), 4.3 (s, 2H). ^{13}C NMR (DMSO-d_6): 169.0, 167.2, 167.1, 166.0, 137.0, 136.0, 135.0, 132.0, 124.5, 124.0, 42.0. Elemental Anal. Calcd for $C_{11}H_7NO_6$: C, 53.02; H, 2.83; N, 5.62. Found: C, 53.04; H, 2.83; N, 5.55.

2.1.2. Synthesis of tetra-aqua bis-4-carboxy-N-phthaloyl glycinato zinc(II) dihydrate $[(\mathbf{L1})_2(H_2O)_4Zn]2H_2O$

To a solution of 4-carboxy-*N*-phthaloylglycine (0.249 g, 1 mmol) in ethanol (20 ml) at room temperature 5 ml of aqueous solution of zinc acetate dihydrate (0.219 g, 1 mmol) was added with constant stirring. The mixture was stirred for 4 h at room temperature and the white precipitate formed was filtered. The precipitate was washed with ethanol (10 ml) and redissolved in water (50 ml), and the solution was left undisturbed. Transparent rectangular crystals were obtained after nine days. Isolated yield: 20%. IR (KBr, cm^{-1}) : 3432(b), 1775(s), 1708(br s), 1592(br s), 1412(s), 1384 (s), 1328 (w), 1199(w), 1111 (s), 965(s), 830 (w), 764 (w), 729(w). ^1H NMR (DMSO-d_6): 8.3 (d, $J = 8.8$ Hz, 1H), 8.2 (s, 1H), 7.9 (d, $J = 7.6$ Hz, 1H), 4.1 (s, 2H). Molar conductance (water) 28.3 Ω^{-1} cm^2 mol^{-1}. Elemental analysis: calcd for $C_{22}H_{24}N_2O_{18}Zn$: C, 39.43; H, 3.59; N, 4.18. Found: C, 39.39; H, 3.70; N, 3.69. Thermogravimetry analysis of the amorphous sample: 1st weight loss of four water molecules at 131 °C: calcd 10.5%, found 9.9%; 2nd weight loss to form ZnO at 460 °C: calcd 88.2%, found 89%.

2.1.3. Synthesis of $[Ni(\mathbf{L2})(py)_3(H_2O)_2]\mathbf{L2}$

To a solution of phthalic anhydride (0.297 g, 2 mmol) in dry methanol (15 ml) magnesium turning (0.024 g, 1 mmol) was added and the mixture was heated at 60 °C for 3 h. During this period the magnesium was dissolved. The reaction mixture was cooled and to this solution a methanolic solution (5 ml) of nickel chloride hexahydrate (0.238 g, 1 mmol) was added. The reaction mixture was stirred at 60 °C for 2 h and filtered. The unreacted residue of magnesium was discarded and from the filtrate methanol was removed to obtain a paste. The paste was dissolved in pyridine (5 ml) to obtain a homogeneous solution. The

Table 1
Crystallographic data for the complexes

Complex	$[(\mathbf{L1})_2(H_2O)_4Zn]2H_2O$	$[Cu_2(\mathbf{L2})_4(H_2O)_2]2H_2O$	$[Ni(\mathbf{L2})(py)_3(H_2O)_2]\mathbf{L2}$
Formulae	$C_{22}H_{28}N_2O_{20}Zn$	$C_{36}H_{36}Cu_2O_{20}$	$C_{33}H_{33}N_3NiO_{10}$
Formula weight	705.83	915.73	690.33
Crystal system	Monoclinic	Triclinic	Triclinic
Temperature (K)	296(2)	296(2)	296(2)
Space group	$P2_1/c$	P-1	P-1
Z	2	1	2
a (Å)	15.6596(15)	8.0203(15)	9.0922(3)
b (Å)	7.2901(7)	10.8089(19)	13.5405(4)
c (Å)	12.7843(13)	12.107(2)	14.6504(5)
α (deg)	90.00	74.624(11)	77.984(2)
β (deg)	100.026(5)	76.394(11)	78.562(2)
γ (deg)	90.00	77.923(10)	72.300(2)
V (Å^3)	1437.2(2)	971.4(3)	1662.89(9)
ρ (calculated) (mg m^{-3})	1.631	1.565	1.379
h, k, l	$-19 \leqslant h \leqslant 22$	$-11 \leqslant h \leqslant 11$	$-12 \leqslant h \leqslant 12$
	$-10 \leqslant k \leqslant 10$	$-15 \leqslant k \leqslant 15$	$-19 \leqslant k \leqslant 19$
	$-16 \leqslant l \leqslant 18$	$-16 \leqslant l \leqslant 17$	$-20 \leqslant l \leqslant 20$
Θ range for data collection,	1.32–30.55°	1.78–30.71°	1.97–30.66°
μ (calculated), mm^{-1}	0.949	1.178	0.644
Absorption correction	None	None	None
Final R indices $[I > 2\sigma]$	$R1 = 0.0283$; $wR2 = 0.0324$	$R1 = 0.0253$; $wR2 = 0.0650$	$R1 = 0.0320$; $wR2 = 0.1054$
R indices [all data]	$R1 = 0.0801$; $wR2 = 0.0837$	$R1 = 0.0298$; $wR2 = 0.0684$	$R1 = 0.0426$; $wR2 = 0.1172$
Gof on F^2	1.037	1.024	0.859

solution on standing gave crystalline product of the complex. Yield: 52%. IR (KBr, cm^{-1}): 3354(br s), 3457(br s), 1726(s), 1629(s), 1491(s), 1414(s), 1306(s), 1260(s), 1127(s), 1081(s), 1040(s), 748(s), 702(s). Visible (acetonitrile) λ_{max} 648 nm ($\varepsilon = 8.98\ M^{-1}\ cm^{-1}$). Molar conductance (water) $13.3\ \Omega^{-1}\ cm^2\ mol^{-1}$. Magnetic moment (RT) 3.04 BM. Elemental Anal. Calcd for $C_{33}H_{33}N_3O_{10}Ni$: C, 57.41; H, 4.78; N, 6.08. Found: C, 57.95; H, 4.88; N, 5.85.

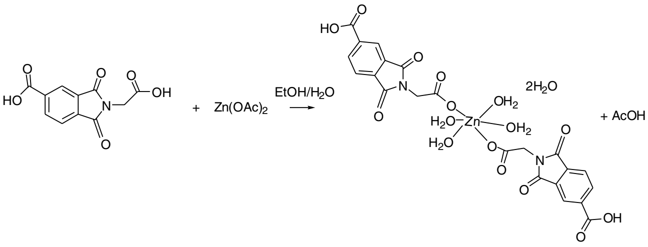
2.1.4. Synthesis of $[Cu_2(\mathbf{L2})_4(H_2O)_2]2H_2O$

Phthalic anhybride (0.297 g, 2 mmol) was dissolved in dry methanol (15 ml). To this magnesium turning (0.024 g, 1 mmol) was added and the mixture was heated at 60 °C for 3 h and during this period the magnesium was dissolved. The reaction mixture was cooled and methanolic solution (5 ml) of cupric chloride dihydrate (0.171 g, 1 mmol) was added. The reaction mixture was stirred at 60 °C for 2 h and filtered. From the filtrate methanol was removed to obtain a paste. The paste was dissolved in a mixed solvent of ethanol and ethylacetate and recrystallised to obtain the complex. Yield: 44%. IR (KBr, cm^{-1}): 3554(br s), 3457(br s), 1726(s), 1629(s), 1491(s), 1414(s), 1306(s), 1260(s), 1127(s), 1081(w), 748(s). Visible (acetonitrile) λ_{max} 691 nm ($\varepsilon = 300\ M^{-1}\ cm^{-1}$). Molar conductance (water) $19.9\ \Omega^{-1}\ cm^2\ mol^{-1}$. Magnetic moment (RT) 1.54 BM. Elemental Anal. Calcd for $C_{36}H_{36}O_{20}Cu_2$: C, 47.21; H, 3.93. Found: C, 47.55; H, 3.94. Thermogravimetry analysis of the crystalline sample: 1st weight loss of four water molecules at temp. 76.3 °C: calcd. 7.54%, found 7.83%; 2nd weight loss to form CuO at 273.7 °C: calcd. 83.4%, found 83.3%.

3. Results and discussion

3.1. 4-Carboxy-N-phthaloylglycinato complex of Zinc(II)

4-Carboxy-*N*-phthaloylglycine (**L1H**) reacts with zinc(II)acetate to give tetra-aquo bis-4-carboxy-*N*-phthaloylglycinato zinc(II) dihydrate (Eq. 1). The structure of the complex is determined by X-ray crystallography (Fig. 1). In this complex the carboxy group attached to the methylene group co-ordinates to the zinc in a monodentate fashion. The 4-carboxy-*N*-phthaloylglycinato ligands are *trans* to each other. The selected bond distances and angles are Zn–O1, 2.097; Zn–O7, 2.086 Å; Zn–O8, 2.115 Å; ⟨O7–Zn–O8, 92.39°; ⟨O1–Zn–O7, 87.95°; ⟨O1–Zn–O8, 96.09°. Accordingly it has a distorted octahedral geometry around the zinc. The intriguing feature of this complex is that, out of the two carboxylic acid groups only one carboxylic acid is deprotonated. This makes the system a metallo-organic hybrid acid. The presence of water molecules in the co-ordination sphere makes it more attractive as these water molecules can be removed easily at 131 °C (vide TG). The porous metallo-organic acids have been of great importance [24].



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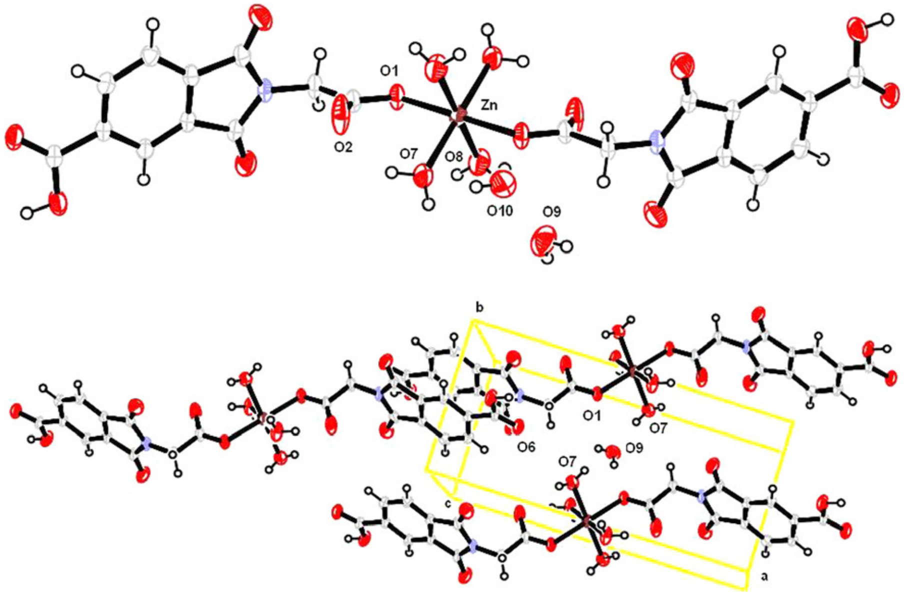


Fig. 1. (Top) Crystal structure of the $[Zn(\mathbf{L1})_2(H_2O)_4]2H_2O$ (thermal ellipsoid with 50% probability). (Bottom) The assembly through π–π and hydrogen-bond interactions.

The 4-carboxy-*N*-phthaloylglycine has carbonyl absorption frequencies in IR at 1787 cm^{-1} and 1724 cm^{-1}, whereas on complexation with zinc these absorptions appear at 1775 cm^{-1} and 1708 cm^{-1}. The proton NMR of the complex has all the peaks of the ligand except at 13.5 ppm due to the carboxylic acid group attached to the methylene group. There is a slight shift in the aromatic protons on complexation. The methylene protons of the ligand appear at 4.3 ppm whereas in the complex they appear at 4.1 ppm. Poor solubility of the complex in common solvent prevented us further NMR study of the complex.

Generally, benzoic acid and their analogues form dimeric structures in the solid state through $R_2^2(8)$ type of hydrogen-bonding interactions between the carboxylic acid groups [18]. Thus, one may expect similar interactions in the case of the zinc complex also, but we did not observe such interactions. However, in this complex the acid group attached to the aromatic ring participates in the formation of self-assembled structure through interaction with the lattice waters and the assemblies are further stabilised by π–π interactions between the aromatic rings (Fig. 1, bottom). The donor–acceptor bond angles and distances are listed in Table 2. The separation distance between the rings that participates in the π–π interaction is 2.238 Å, which is well within the permissible limit [25]. The molar conductance of the complex shows it to be non-ionic in water.

In the crystal lattice, the co-ordinated water molecules are in two different environments. One set of water molecules has chelate type of interaction with carbonyl group of carboxylate. The other set is hydrogen bonded to lattice water. The non-coordinating carboxylic acid group is hydrogen bonded to the carbonyl oxygen of a carboxylate group of another molecule. This results in the formation of self-assembled structure. Two types of environments for co-ordinated water molecules are shown in Fig. 2. The segment **A** shows participation of co-ordinated water molecules in forming six-membered chelate like structure through H-bonding interactions, and the segment **B** shows the H-bonding of co-ordinated water molecule with the lattice water and the free OH group of carboxylic acid group of another molecule. Similar kind of observation was also made by us in the case of zinc – tetra aquo bis-*N*-phthaloylglycinato zinc(II) dihydrate complex [16]. From our foregoing discussion it is clear that the difference in reactivity of two carboxylic acid groups in different environment can lead to selective deprotonation.

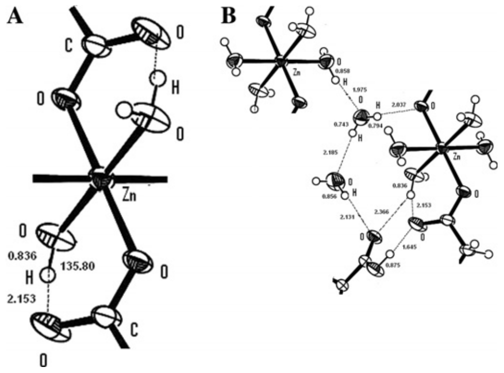


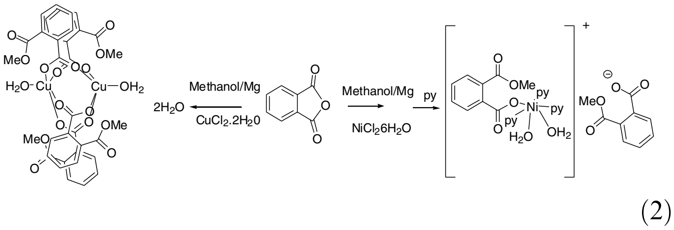
Fig. 2. Segment of the zinc complex showing different environments around coordinated water.

Table 2
Donor–acceptor bond distances (Å) and bond angles in $[Zn(\mathbf{L1})_2(H_2O)_4]2$-$H_2O$ to form three dimensional network

$d_{D\cdots A}$	Distance (Å)	Symmetry
O9···O1	2.823	$1-x, 1/2+y, 1/2-z$
O7···O9	2.759	$1-x, 1/2+y, 1/2-z$
O9···O10	2.900	$1-x, -1/2+y, 1/2-z$
O8···O2	2.813	$x, -1/2-y, -1/2+z$
O10···O4	2.939	$x, -1/2+y, 1/2-z$
O5···O10	2.924	$-1+x, 1/2+y, 1/2-z$
O2···O6	2.520	$2-x, -1/2+y, 1/2-z$
⟨O2–H···O6	177.65	

3.2. 2-Carbomethoxy benzoato complexes of nickel(II) and copper(II)

It is also important to know the effect of a neighboring group/s in carboxylate binding. For this purpose, we have studied the reaction of 2-carbomethoxy benzoic acid with zinc(II) acetate. From this reaction, we obtained an insoluble product, which we could not characterise completely. However, an alternative procedure allowed us to prepare nickel and copper complex of 2-carbomethoxy benzoate. In this procedure, nickel chloride or copper chloride with phthalic anhydride in methanol was reacted with magnesium. The role of magnesium is to generate the methoxide anion, which in turn opens the ring of phthalic anhydride to give magnesium salt of 2-carbomethoxy benzoic acid. Such reaction of nickel(II) chloride followed by crystallisation from pyridine (py) (right side of Eq. 2) gave a nickel complex $[Ni(\mathbf{L2})(py)_3(H_2O)_2]\mathbf{L2}$ (where $\mathbf{L2}$ = 2-carbomethoxy benzoate).



(2)

The complex has a distorted octahedral geometry around the Ni(II). Two pyridine ligands occupy the axial positions (top, Fig. 3). In this complex, one 2-carbomethoxy benzoate is mono-dentate and the other is ionic. The monodentate carboxylate, a pyridine and two water molecules lie in one plane. The two water molecules are *cis* to each other and are hydrogen bonded to an ionic carboxylate. One coordinated water molecule is also hydrogen bonded to carbonyl group (Fig. 3 bottom). The selected donor–acceptor bond angles and bond distances in the assembly are given in Table 3. The ester group of the ligand

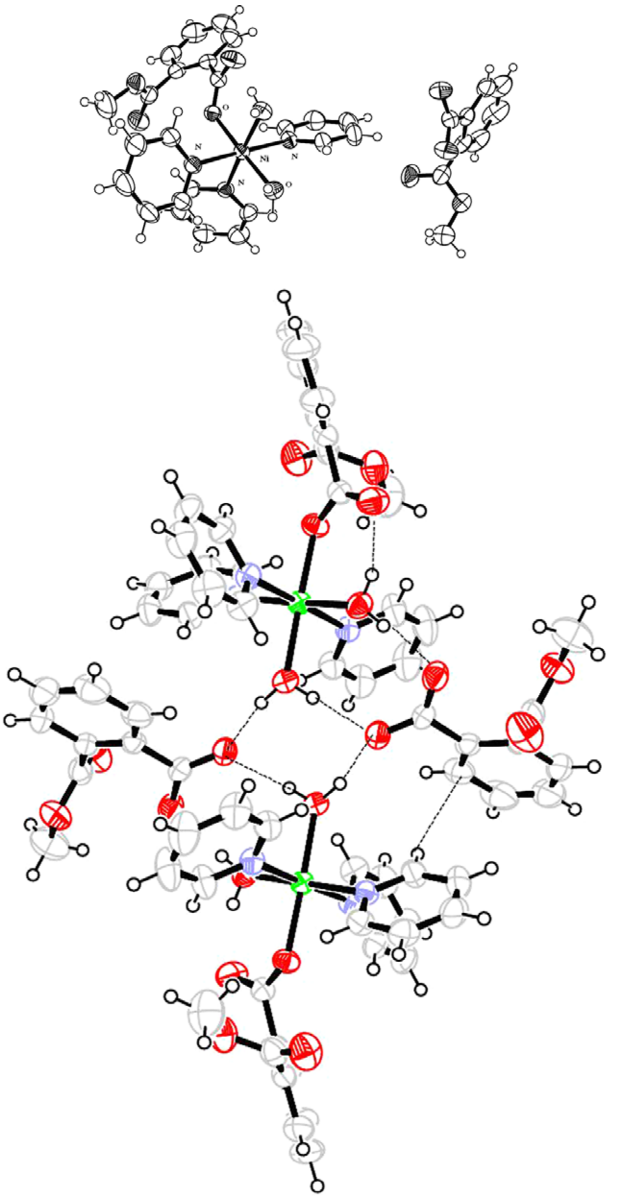


Fig. 3. (Top) Structure of [Ni(**L2**)(py)$_3$(H$_2$O)$_2$]**L2**(thermal ellipsoid with 50% probability). (Bottom) Weak C–H$\cdots\pi$ interactions and H-bonding interactions for formation of self assembly.

Table 3
Donor–acceptor bond distances (Å) and bond angles (°) in [Ni(**L2**)(py)$_3$(H$_2$O)$_2$]**L2**

$d_{\mathrm{D}\cdots\mathrm{A}}$	Distance (Å)	Angles (°)
O6$\cdots$O13	2.64	174.34
O6$\cdots$O14	2.73	175.34
O5$\cdots$O14	2.73	175.34
O6$\cdots$O13	2.64	174.37

appearing at 1731 cm^{-1} is unaffected in the complex, which shows that the ester group does not participate in the coordination. There are also sharp absorptions from coordinated carboxyl group at 1639 cm^{-1} and due to the free carboxylic acid at 1603 cm^{-1}.

The complex has a broad and sharp absorption at 3086 cm^{-1} due to O–H stretching of water molecules. The free carboxylates are held in an assembly formed through hydrogen bonding and C–H$\cdots\pi$ interactions as illustrated in Fig. 3 (bottom). The donor–acceptor distances are listed in Table 2. The molar conductance of the complex in solution is low and suggests it to be non-ionic, which may be due to strong ion-pair formation. The compound has visible absorbance at 648 nm, which is assigned to $^3A_2 \rightarrow {}^3T_1$ transition. The compound has a magnetic moment of 3.04 BM which corresponds to two unpaired electrons.

It is found that the complex of 2-carbomethoxy benzoate with copper(II) has a dimeric structure which resembles the structure of copper(II) acetate monohydrate [22]. However, the Cu$_2$(**L2**)$_4$(H$_2$O)$_2$]2H$_2$O has two water molecules at axial positions. The Cu–O bond lengths for the coordinated water with Cu^{2+} are 2.168 and 2.142 Å (Fig. 4). In this complex, the copper–copper distance is 2.624 Å, which is comparable to the Cu$\cdots$Cu distance in dimeric copper(II) acetate [22] which is 2.614 Å. The magnetic moment of the complex is 1.54 BM which is also comparable to the magnetic moment of copper(II) acetate dimer. But the interesting feature of the complex is the formation of self-assembled structure as illustrated in Fig. 5. In the lattice, the water of crystallisation is held by hydrogen bonding between coordinated water as well as by carbonyl groups of esters. The donor–acceptor bond length and angles for hydrogen bonding that are responsible for assembly formation are listed in Table 4.

The IR-spectra of the complex show absorption at 1726 cm^{-1} due to the ester group as compared to the ligand

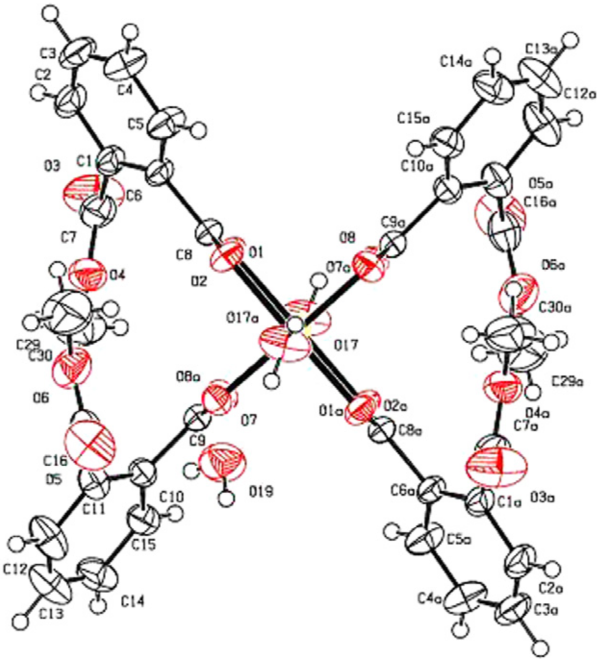


Fig. 4. Structure of [Cu$_2$(**L2**)$_4$(H$_2$O)$_2$]2H$_2$O (thermal ellipsoids drawn to 50% probability).

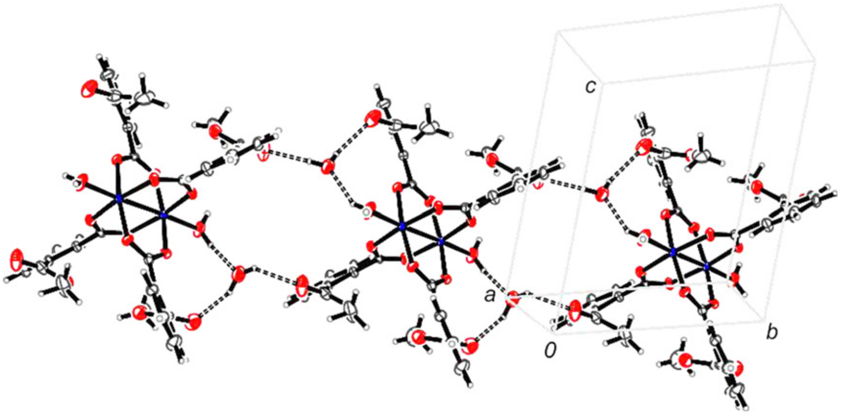


Fig. 5. Hydrogen bonding network in the lattice of $[Cu_2(\mathbf{L2})_4(H_2O)_2]2H_2O$.

Table 4
Donor–acceptor bond distances (Å) and angles (°) in $[Cu_2(\mathbf{L2})_4(H_2O)_2]2H_2O$

$d_{D\cdots A}$	Distance (Å)	Angles (°)
O5···O19 $[x-1, y, z]$	2.89	152.94
O17···O19 $[1-x, 1-y, -z]$	2.72	168.92
O3···O19 $[1-x, 1-y, -z]$	2.83	176.78

that occurs at 1731 cm^{-1}. It has also strong absorption at 1629 cm^{-1} due to the carboxylate groups. The OH stretching for water appearing at 3554 cm^{-1} 3457 cm^{-1} shows that there are two different types of environment for water molecules. The complex absorbs at 691 nm and this absorption is assigned to $^2E \rightarrow {}^2T_2$ transition for the complex. The low molar conductance observed in solution for the complex suggests it is of non-ionic character.

In conclusion, a metallo-organic hybrid carboxylic acid is prepared in the form of bis-4-carboxy-*N*-phthaloylglycinato zinc(II) dihydrate. The complex forms self-assembly via π–π interaction and hydrogen bonding. Self-assemblies of a monomeric nickel complex of 2-carbomethoxy benzoate and a dimeric copper complex of 2-carbomethoxy benzoate are discussed.

4. Supplementary material

Supplementary data on the crystal structures are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, on request, quoting the deposition number(s) CCDC 285990, 285992, and 285994.

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