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Journal of Molecular Structure 796 (2006) 107-113

Journal of MOLECULAR STRUCTURE

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# Construction of one- and two-dimensional coordination polymers using ditopic imidazole ligands

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Received 31 January 2006; received in revised form 24 February 2006; accepted 24 February 2006 Available online 3 May 2006

#### Abstract

A series of ditopic imidazole functionalised ligands have been investigated for their propensity to form one- and two-dimensional coordination polymers with a range of metals. We show that ditopic imidazole ligands have the versatility to be exploited in the formation of a variety of coordination polymers.

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Keywords: Coordination polymer; Imidazole; Crystal structure analysis; Crystal engineering; Topology

#### 1. Introduction

Owing to their fascinating structural motifs and the promise of new functional materials for diverse applications, coordination polymers have attracted significant attention over the last two decades [1-5]. The formation of one-, two- and threedimensional frameworks relies on the use of ditopic or higher ordered ligands and, in this context, pyridyl and carboxylate functional groups are abundantly represented in the literature. Considering their high affinity for metals and their relative ease of functionalisation, imidazole functionalised ligands have attracted surprisingly little attention to date [6]. Nevertheless, the few existing examples of coordination polymers involving imidazole derivatised ligands feature several interesting structural motifs and topologies, which include polyrotaxanes, pseudo-polyrotaxanes, two-dimensional Borromean frameworks, poly-metallocage structures and adamantane-like  $6^6$ nets [7-13]. The overall objective of investigating the formation of new topologies is to ultimately achieve the rational design and construction of targeted coordination networks. Indeed, some success has already been achieved with the design of porous materials that utilize imidazole based ligands [12–16]. In this contribution, we illustrate the ease of functionalisation of the imidazole group to yield three structurally related ligands (Scheme 1), which have been used to construct a range of interesting topologies.

## 2. Experimental section

All commercially available chemicals were of reagent grade and were used as received, without further purification. The ligands 1,4-bis(2-methylimidazol-1-ylmethyl)benzene (1), 1,4bis(benzimidazol-1-ylmethyl)benzene (2) and 4,4'-bis(benzimidazol-1-ylmethyl)biphenyl (3) were synthesised by the  $S_N2$ reaction of the imidazole moieties with the appropriate arenyl dichloride spacer.

Single crystal X-ray diffraction data were recorded using a Bruker SMART APEX diffractometer equipped with graphitemonochromated Mo K $\alpha$  radiation ( $\lambda$ =0.71073 Å). Empirical absorption corrections were applied using sADABS [17]. Structures were solved using sHELXS 97 [18] and all ordered non-hydrogen atoms were refined anisotropically by fullmatrix least-squares on  $F^2$  using sHELXL-97 under the X-Seed environment [19–20]. Where feasible, hydrogen atoms were either placed in calculated positions or located in difference electron density maps. Crystal data are given in Table 1. Hydrogen bond and coordination sphere geometric parameters are available in supplementary material and CIF files. CCDC reference numbers: 296902–296908. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre,

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Scheme 1.

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## 3. Results and discussion

# 3.1. $[Cu(NO_3)_2(H_2O)(1)_{1.5} \cdot 4H_2O]_n$ (4)

Crystals suitable for X-ray diffraction analysis were obtained by the reaction of  $Cu(NO_3)_2 \cdot 3H_2O$  with 1 (1:4 molar ratio) in MeOH, followed by slow evaporation of the solvent. The asymmetric unit consists of a  $Cu^{2+}$  ion and two water molecules, all situated on a twofold rotation axis at 1/2, *y*, 3/4, one and a half ligands 1, one water molecule on a general

Table 1			
Data collection and	final refinement	parameters	for 4-10

position and a nitrate anion disordered over two positions. The copper cation is five-coordinate with square pyramidal geometry defined by one apical water molecule and four equatorial ligands 1. A one-dimensional coordination polymer is formed parallel to [101] with ligand 1 assuming two distinctive roles in connecting successive copper ions (Fig. 1). One of the ligands is coordinated directly to two copper centres by means of its two imidazole moieties (i.e. Cu-1-Cu). A further two indirect Cu...Cu connections are present by virtue of hydrogen bonded water bridges (i.e. Cu-H<sub>2</sub>O···H<sub>2</sub>O···H- $(O(coordinated) \cdots O = 2.775(5) \text{ Å} and O \cdots N =$ Cu) 2.834(6) Å) where each ligand is only coordinated to one metal centre via one of its imidazole groups, while the uncoordinated imidazole group accepts a hydrogen bond from a lattice water molecule which, in turn, accepts a hydrogen bond from a coordinated water molecule on an adjacent copper ion. Taken together, the two indirect (...Cu-H<sub>2</sub>O...H<sub>2</sub>O...H<sub>2</sub>O...I- $Cu-H_2O\cdots H_2O\cdots I-$ ) linkages can be thought of as a 34membered ring through, which the directly coordinated exobidentate ligand is threaded and it may therefore be appropriate to refer to this arrangement as a pseudo-polyrotaxane.

#### 3.2. $[Co(NCS)_2(CH_3OH)(1)_{1.5})]_n$ (5)

Single crystals of **5** were obtained by slow evaporation of a methanolic solution of  $CoCl_2 \cdot 6H_2O$ , KSCN and **1** in a 1:2:4 molar ratio. The asymmetric unit consists of one  $Co^{2+}$  cation, one and a half ligands of **1**, two thiocyanate counter ions and one methanol molecule. The cobalt ion is in an octahedral coordination environment consisting of three ligands of **1**, two *N*-coordinated thiocyanate anions and a coordinated methanol molecule. Two ligands **1** are *trans* to one another and *cis* to the

	4	5	6	7	8	9	10
Empirical formula	C48 H62 Cu	C27 H40 Co	C25 H27 Ag	C44 H36 Cd	C20 H28 Cu	C26 H32 Cu	C54 H56 Cu2
•	N14 O10	N8 O S2	F3 N6 O3 S	Cl2 N8	N4 O6	N4 O8	N7 O10
Formula weight	1058.66	612.70	656.46	860.11	484.01	592.10	1090.14
<i>T</i> (K)	100(2)	298(2)	100(2)	100(2)	100(2)	100(2)	100(2)
Crystal system	Monoclinic	Monoclinic	Trigonal	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	C2/c	P21/n	P-3	P21/n	P21/c	P21/c	C2/c
a (Å)	15.977(2)	11.1229(8)	14.2551(11)	11.5035(14)	9.3730(10)	9.7515(16)	42.215(6)
b (Å)	12.4892(18)	10.0174(7)	14.2551(11)	13.3651(16)	9.7008(10)	16.203(3)	15.666(2)
<i>c</i> (Å)	25.713(4)	25.8715(18)	7.7890(12)	11.7868(14)	13.3403(14)	17.360(3)	15.641(2)
$\alpha^{\circ}$	90	90	90	90	90	90	90
$\beta^{\circ}$	100.952(3)	90.2590(10)	90	90.452(2)	107.908(2)	97.432(3)	100.354(3)
$\gamma^{\circ}$	90	90	120	90	90	90	90
Ζ	4	4	2	2	2	4	8
$V(Å^3)$	5037.3(12)	2882.6(4)	1370.7(3)	1812.1(4)	1154.2(2)	2719.8(8)	10175(3)
$D (g/cm^3)$	1.396	1.419	1.590	1.576	1.393	1.446	1.423
Absorption coefficient $(mm^{-1})$	0.506	0.777	0.870	0.797	0.988	0.859	0.902
Final $R$ indices $[I > 2 \text{sigma}(I)]$ :	0.0749	0.0633	0.0551	0.0365	0.0430	0.1211	0.0821
$R_1, wR_2$							
	0.2024	0.1458	0.1279	0.0815	0.1111	0.2816	0.1928
<i>R</i> indices (all data): $R_1$ , $wR_2$	0.1130	0.0916	0.0630	0.0427	0.0538	0.1797	0.1276
	0.2326	0.1601	0.1324	0.0843	0.1174	0.3244	0.2185
Goodness-of-fit on $F^2$	1.027	1.040	1.090	1.036	1.022	1.051	1.038
Largest diff. peak and hole (e $\text{\AA}^{-3}$ )	0.953	0.776	1.272	1.150	0.468	4.020	1.484
	-0.569	-0.638	-0.951	-0.403	-0.197	-1.976	-0.566



Fig. 1. Perspective view of the one-dimensional coordination polymer in **4**. The 34-membered rings, each incorporating four hydrogen bonds, are shown in red. These rings are threaded by directly coordinated ditopic ligands (yellow). All non-metal atoms are shown in capped-stick representation (for interpretation of the reference to colour in this legend, the reader is referred to the web version of this article).

third ligand. Owing to this arrangement of the three ditopic ligands, the metal centres act as three-connected nodes and a two-dimensional (6,3) distorted honeycomb coordination network is formed (Figs. 2 and 3) parallel to (101). The rings are large and corrugated and thus result in the occurrence of threefold parallel interpenetration (Fig. 3).

The approximately linear NCS<sup>-</sup> anions are both tilted with respect to their respective Co–N vectors with Co–N–C angles of ca. 160°. The coordinated methanol molecule is similarly tilted with a Co–O–C angle of approximately 125°. One of the thiocyanate sulphur atoms accepts a hydrogen bond from a coordinated methanol hydroxyl group (S…O=3.225 Å) of another (interpenetrated) layer and this relatively rare hydrogen bond helps to bind the triply interpenetrated layers to one another.

# 3.3. $[Ag(1)(CF_3SO_3)]_n$ (6)

Crystals were grown by slow evaporation of a methanolic solution of **1** and  $Ag(CF_3SO_3)$  in a 1:1 molar ratio. X-ray structural analysis reveals the formation of a Borromean weave (Fig. 4) remarkably similar to that of a structure recently reported by us ({ $[Ag_21_3](BF_4)_2$ }, **6a**) [10]. The only difference in composition between **6** and **6a** is that, in the latter, the anion

is  $BF_4^-$  instead of  $CF_3SO_3^-$ . Although the overall topology of the Borromean weave has already been described in detail, it is interesting to now discuss the subtle influence exerted on the structure by the anion. Both anions possess threefold rotation symmetry, but the most obvious difference between them is that the triflate anion is somewhat larger than the tetrafluoroborate anion. The most notable difference between the two structures is that the Ag...Ag separation in 6a is 3.0619(4) Å while the corresponding distance in **6** is 3.225(1) Å. Since the anions are stacked in columns along [001], it is tempting to presume that their stacking interval is responsible for the adjustment of the argentophilic interactions. However, the anions are not within van der Waals contact of one another along [001] in either structure. In both structures, three imidazole groups are situated about each silver ion in a propeller-like fashion. Owing to steric effects, the imidazole groups are required to rotate about the Ag-N vector such that the methyl group of one imidazole moiety should be either above or below the plane of the neighbouring imidazole ring. In 6a, the methyl groups are oriented towards the outer surface of the Borromean layer while they are oriented inwards in 6. These two possible orientations of the imidazole rings about the silver ion have a remarkable affect on the size of the triangular cavity formed between the three components of the



Fig. 2. Ball-and-stick perspective view of 5 showing six three-connected nodes that form the large rings of a (6,3) network.



Fig. 3. The parallel triple interpenetration of the two-dimensional (6,3) nets in **5**. Individual nets, shown in red, yellow and blue, are idealised by representing the metal...metal connection via ligand **1** as straight pipes (for interpretation of the reference to colour in this legend, the reader is referred to the web version of this article).

Borromean weave (Fig. 5). When the methyl groups are oriented outwards, the cavity becomes smaller and packing interactions with smaller anions would thus favour this conformation of the complex. However, enlargement of the cavity by rotation of the imidazole ring such that the methyl groups are directed inwards occurs at the expense of the Ag $\cdots$ Ag interactions. Therefore, it appears that accommodation of larger anions and the formation of stronger argentophilic interactions are competing effects.

## 3.4. $[CdCl_2(2)_2]_n(7)$

Crystals were grown from a solution of 2 in dichloromethane, layered with a solution of  $CdCl_2 \cdot 2.5H_2O$  in methanol. The asymmetric unit consists of a cadmium cation situated on an inversion centre, one chloride anion and one ligand 2. The cadmium ion is in an octahedral coordination





Fig. 5. Perspective views of **6a** (top) and **6** (bottom) along [001] showing the triangular voids normally occupied by the anions, which have been omitted for clarity. The size and shape of the void spaces are influenced by the orientations of the 2-methyl groups (red) of the imidazole moieties (for interpretation of the reference to colour in this legend, the reader is referred to the web version of this article).



Fig. 6. Projections of **7** along [101] showing the formation of a two-dimensional coordination polymer in capped-stick (top) and space filling representations (bottom).

environment with two axial chloride ions and four ligands of **2** in equatorial positions. Two adjacent benzimidazole groups are positioned with their benzo moieties on one side of the equatorial plane while the remaining two benzo moieties are on the other side of the plane. This alternative to a propeller-like arrangement also serves to alleviate steric effects and a neutral two-dimensional (4,4) net is formed (Fig. 6). The ligands assume a conformation that occupies space efficiently and no solvent molecules are present in the structure. Owing to the abundance of aromatic groups, the two-dimensional layers are dominated by edge-to-face  $\pi \cdots \pi$  interactions although

adjacent layers associate only by means of van der Waals interactions [21].

# 3.5. $[Cu(CH_3COO)_2(1)_2]_n$ (8)

Crystals were grown by slow evaporation of 1 and (CH<sub>3</sub>COO<sub>2</sub>)·H<sub>2</sub>O in a 3:2 molar ratio. X-ray analysis reveals the formation of simple, symmetry-related one-dimensional polymeric chains (Fig. 7) parallel to [1-10] and [110]. The copper ion is in a square planar environment with two ligands 1 coordinated trans to one another (Table 2). The remaining two coordination sites are occupied by monodentate acetate anions. We presume that formation of the paddle-wheel arrangement or bidentate coordination of acetate is sterically hindered by the imidazole methyl groups. The non-coordinated oxygen atom of the acetate ligand accepts a hydrogen bond from a water molecule situated in the lattice. The water molecule also donates a hydrogen bond to the coordinated acetate oxygen atom of an adjacent one-dimensional strand. A series of onedimensional strands running parallel to [1-10] can be thought of as forming a two-dimensional layer. The next layer along [001] consists of one-dimensional strands running parallel to [110] and thus adjacent layers of strands criss-cross one another at an angle of approximately 92°. Thus the hydrogen bonded water bridges stitch adjacent layers to one another.

# 3.6. $[Cu(CH_3COO)_2(H_2O)(2)_2 \cdot 3H_2O]_n$ (9)

Crystals were obtained from a solution of 2 in chloroform, layered with a solution of copper acetate monohydrate in acetonitrile. The copper ion is in a square pyramidal environment with two ditopic ligands 2 trans to one another to yield a one-dimensional chain (Fig. 8 and Table 2). The remaining trans coordination sites are occupied by two monodentate acetate anions in a manner similar to that of 8. A water molecule is coordinated to the apical site of the copper ion. The square pyramids of adjacent metal centres are oriented in an up-down fashion. All of the strands in the structure are aligned parallel to [-101]. An extensive network of hydrogen bonds serves to bind the strands together (Fig. 9). The coordinated water molecule donates one hydrogen bond to a water molecule situated in the lattice, which, in turn, donates two hydrogen bonds to uncoordinated acetate oxygen atoms of two separate chains. A second lattice water molecule donates a hydrogen bond to one coordinated acetate oxygen atom as well



Fig. 7. Capped-stick representation of the one-dimensional polymer in eight composed of square planar (shown in yellow) copper centres linked to one another by ligand 1 (for interpretation of the reference to colour in this legend, the reader is referred to the web version of this article).

Table 2	
Coordination geometric parameters for copper acetate structures 8, 9 and 10 (Å,	°)

8	9	10-Cu(1)	10-Cu(2)
Cu(1)–N(3)#1 1.9756(18)	Cu(1)–N(3)#1 1.982(6)	Cu(1)-O(2)#1 1.964(4)	Cu(2)–O(5) 1.962(5)
Cu(1)-N(3) 1.9756(18)	Cu(1)–N(2) 1.991(6)	Cu(1)–O(1) 1.971(4)	Cu(2)–O(9) 1.978(5)
Cu(1)-O(1) 1.9928(17)	Cu(1)–O(8) 1.995(5)	Cu(1)-O(3) 1.997(4)	Cu(2)–N(1) 1.987(6)
Cu(1)-O(1)#1 1.9928(17)	Cu(1)–O(1) 1.995(6)	Cu(1)-N(8) 2.137(4)	Cu(2)-N(11) 1.990(5)
	Cu(1)–O(5) 2.427(8)		
N(3)#1-Cu(1)-N(3) 180.00(12)	N(3)#1-Cu(1)-N(2) 171.2(3)	O(2)#1-Cu(1)-O(1) 167.50(15)	O(5)-Cu(2)-O(9) 88.9(2)
N(3)#1-Cu(1)-O(1) 91.28(8)	N(3)#1-Cu(1)-O(8) 90.6(2)	O(2)#1-Cu(1)-O(4)#1 89.06(18)	O(5)-Cu(2)-N(1) 161.9(2)
N(3)-Cu(1)-O(1) 88.72(8)	N(2)-Cu(1)-O(8) 89.9(2)	O(1)-Cu(1)-O(4)#1 89.45(17)	O(9)-Cu(2)-N(1) 92.0(2)
N(3)#1-Cu(1)-O(1)#1 188.72(8)	N(3)#1-Cu(1)-O(1) 88.0(3)	O(2)#1-Cu(1)-O(3) 89.25(17)	O(5)-Cu(2)-N(11) 91.73(19)
N(3)-Cu(1)-O(1)#1 191.28(8)	N(2)-Cu(1)-O(1) 91.0(2)	O(1)-Cu(1)-O(3) 89.57(16)	O(9)-Cu(2)-N(11) 164.4(2)
O(1)-Cu(1)-O(1)#1 1180.00(9)	O(8)-Cu(1)-O(1) 176.1(3)	O(4)#1-Cu(1)-O(3) 167.73(16)	N(1)-Cu(2)-N(11) 92.2(2)
C(6)-O(1)-Cu(1) 103.98(14)	N(3)#1-Cu(1)-O(5) 97.1(3)	O(2)#1-Cu(1)-N(8) 100.34(16)	
	N(2)-Cu(1)-O(5) 91.7(3)	O(1)-Cu(1)-N(8) 92.16(16)	
	O(8)-Cu(1)-O(5) 86.8(3)	O(4)#1-Cu(1)-N(8) 98.46(16)	
	O(1)-Cu(1)-O(5) 97.0(3)	O(3)-Cu(1)-N(8) 93.79(16)	
		O(2)#1-Cu(1)-Cu(1)#1 85.10(11)	
		O(1)-Cu(1)-Cu(1)#1 82.41(11)	
		O(4)#1-Cu(1)-Cu(1)#1 85.17(12)	
		O(3)–Cu(1)–Cu(1)#1 82.58(11)	
		N(8)-Cu(1)-Cu(1)#1 173.47(12)	

Cu(1) refers to the paddle–wheel coordination sphere and Cu(2) refers to the square planar coordination sphere of 10. Symmetry transformations used to generate equivalent atoms for structure: 8: #1 x - 1, -y + 3/2, z + 1/2 #2 x + 1, -y + 3/2, z - 1/2; 9: #1 x - 1, -y + 3/2, z + 1/2 #2 x + 1, -y + 3/2, z - 1/2; 10: #1 -x + 1/2, -y + 1/2, -z + 2 #2 -x, y, -z + 1/2.



Fig. 8. Capped-stick representation of the one-dimensional molecular strand in **9**. The copper cations are in a square pyramidal coordination environment (in yellow). Successive square-based pyramids are canted at an angle of ca. 135° relative to one another (for interpretation of the reference to colour in this legend, the reader is referred to the web version of this article).

as to a third lattice water molecule. The latter donates a hydrogen bond to a coordinated acetate oxygen atom and the either donates or accepts a hydrogen bond to or from a symmetry related instance of itself.

# 3.7. $[Cu_2(CH_3COO)_4(3)_{1.5} \cdot CH_3CN \cdot 2CH_3OH]_n$ (10)

Crystals were prepared by the combination of equimolar solutions of metal salt (copper acetate monohydrate in acetonitrile) and ligand **3** (methanol), followed by diffusion of ether into the mixture. X-ray analysis reveals the unusual formation of one-dimensional strands containing two different coordination geometries (Fig. 10). The well-known coordination of four acetate anions by two copper cations (i.e. the paddle–wheel arrangement) occurs at one node (Table 2). The apical positions of the two copper ions are each occupied by benzimidazole groups of ligand **3**. The other end of the ditopic ligand is coordinated to a copper ion in a distorted square planar environment (Table 2). Another ligand **3** is coordinated to this metal in one of the *cis* positions while the other *cis* position, as well as the *trans* position are occupied by



Fig. 9. Capped-stick representation of the hydrogen bonding network involving water molecules in 9. A two-dimensional layer is formed. Contiguous hydrogen bonded water tetramers are shown in yellow and the water molecules of hydrogen bonded rings, which include non-coordinated acetate oxygen atoms, are shown in blue. Ligand 2 has been omitted for clarity (for interpretation of the reference to colour in this legend, the reader is referred to the web version of this article).



Fig. 10. Capped-stick representation of the one-dimensional coordination polymer of compound **10**. Note that the paddle–wheel motif forms a linear coordination node while the distorted square planar moiety forms a bent coordination node.

monodentate acetate anions. The other end of the *cis* ligand **3** is coordinated to a symmetry-related instance of the square planar copper ion, which is in turn linked via another ditopic ligand **3** to a paddle–wheel motif. The one-dimensional chain described above forms an approximate sine wave parallel to [101] with the paddle–wheel motifs situated on the horizontal axis of the wave. At the square planar copper node, each of the non-coordinated acetate oxygen atoms accepts a hydrogen bond from a methanol solvent molecule situated in the lattice (donor—acceptor distances of 2.90(2) and 2.83(2) Å).

## 4. Conclusion

We have shown that ditopic imidazole-based ligands can have rich coordination chemistry for the purposes of constructing a range of coordination polymers. These ligands can be synthesised with relative ease and functionalised with bulky substituents on the imidazole rings and also linked by means of a variety of spacer groups. Owing to the versatile ability of imidazole ligands to bind a range of metal ions, it is envisioned that further studies will facilitate the construction of interesting new coordination polymeric materials.

#### Acknowledgements

Financial support was provided by the National Research Foundation of South Africa and LD thanks the Claude Harris Leon Foundation for a postdoctoral fellowship.

#### Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2006.02.059.

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