metal-organic compounds

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Coordination-directed one-dimensional coordination polymers generated from a new oxadiazole bridging ligand and HgX_2 (X = Cl, Br and I)

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A new 1,3,4-oxadiazole bridging bent organic ligand, 2,5-bis{5methyl-2-[(4-pyridyl)methoxy]phenyl}-1,3,4-oxadiazole, C₂₈H₂₄N₄O₃, L, has been used to create three novel onedimensional isomorphic coordination polymers, viz. catenapoly[[[dichloridomercury(II)]-µ-2,5-bis{5-methyl-2-[(4-pyridyl)methoxy]phenyl]-1,3,4-oxadiazole] methanol monosolvate], {[HgCl₂(C₂₈H₂₄N₄O₃)]·CH₃OH}_n, catena-poly[[[dibromidomercury(II)]-µ-2,5-bis{5-methyl-2-[(4-pyridyl)methoxy]phenyl}-1,3,4-oxadiazole] methanol monosolvate], {[HgBr₂(C₂₈H₂₄- N_4O_3]·CH₃OH_n, and *catena*-poly[[[diiodidomercury(II)]- μ -2,5-bis{5-methyl-2-[(4-pyridyl)methoxy]phenyl}-1,3,4-oxadiazole] methanol monosolvate], $\{[HgI_2(C_{28}H_{24}N_4O_3)] \cdot CH_3 OH_n$. The free L ligand itself adopts a *cis* conformation, with the two terminal pyridine rings and the central oxadiazole ring almost coplanar [dihedral angles = 5.994 (7) and 9.560 (6)°]. In the Hg^{II} complexes, however, one of the flexible pyridylmethyl arms of ligand L is markedly bent and helical chains are obtained. The HgII atom lies in a distorted tetrahedral geometry defined by two pyridine N-atom donors from two L ligands and two halide ligands. The helical chains stack together via interchain π - π interactions that expand the dimensionality of the structure from one to two. The methanol solvent molecules link to the complex polymers through O- $H \cdots N$ and $O - H \cdots O$ hydrogen bonds.

Comment

Self-assembly of organic ligands and inorganic metal ions is one of the most efficient and widely utilized approaches towards the construction of metal–organic coordination polymers or supramolecular complexes (Braga *et al.*, 2003; Kitagawa *et al.*, 2004; Long & Yaghi, 2009; Ma *et al.*, 2010). Helical assemblies constructed from chiral or achiral reagents are prevalent in biology and pharmacology, and also play a key role in supramolecular recognition, replication and catalysis (Albrecht, 2001). Recently, significant developments have been made in artificial supramolecular architectures with helicity *via* coordination (Giuseppone *et al.*, 2006; Du *et al.*, 2009) or hydrogen-bonding interactions (Lee *et al.*, 2007).

During the past decade, the design and construction of rigid and flexible organic ligands bridged by 1,3,4-oxadiazole have been pursued, due to their diversity in coordination chemistry and model applications in functional materials (Jabbour *et al.*, 2002; Hughes & Bryce, 2005; Du *et al.*, 2010). It is well known that π - π interactions play an important role in determining the arrangement of supramolecular compounds with these ligands (Das *et al.*, 2010).



So far, various organic ligands have been used for molecular helical building blocks, but 1,3,4-oxadiazole-based bent organic ligands as a helical component have remained rare until recently. Previously, a study of the Ag^I coordination chemistry of 2,5-bis[3-(3-pyridylcarbonyl)phenyl]-1,3,4-oxadiazole (L4; Dong et al., 2006) found that L4 and Ag^I can be employed as angular directional components to give rise to a helical skeleton, which could be considered as an alternative rational approach to accessing helical metal-organic polymers. As part of our systematic investigation of self-assembly based on bent ligands of this type, we have synthesized a new 1,3,4oxadiazole bridging bent ligand, 2,5-bis{5-methyl-2-[(4-pyridyl)methoxy]phenyl]-1,3,4-oxadiazole, L or (I), and three novel metal-organic frameworks with the same architectures and topologies, [(HgLCl₂)·CH₃OH]_n, (II), [(HgLBr₂)·CH₃- OH_{n} , (III), and $[(HgLI_2) \cdot CH_3OH]_n$, (IV), the structures of which we report here.



Figure 1 The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

Compound (I) (Fig. 1) crystallizes in the monoclinic space group $P2_1/n$ with a single independent molecule in the asymmetric unit. The two terminal pyridylmethyl groups adopt a *cis* conformation with respect to the central bridging oxadiazole group. The two terminal pyridine rings and the central oxadiazole ring of (I) are almost coplanar, with dihedral



Figure 2

The molecular structure of (II), with displacement ellipsoids drawn at the 30% probability level. Hydrogen bonds are shown as dashed lines. Compounds (III) and (IV) are isostructural with (II). [Symmetry codes: (i) x, y, z + 1; (ii) x, y, z - 1.]

angles between the planes of the pyridine and oxadiazole rings of 5.994 (7) and 9.560 (6)°. Compared with L4 (Dong *et al.*, 2006), the methylene group in (I) is more flexible than the carbonyl group in L4, making (I) more attractive for the design of novel supramolecular complexes.

The isomorphous compounds (II), (III) and (IV) crystallize in the triclinic space group $P\overline{1}$, with one Hg^{II} atom, one L ligand, two halide ligands and one methanol solvent molecule in the asymmetric unit. The Hg^{II} atom is coordinated by two pyridine N atoms [N3 and N4ⁱ; symmetry code: (i) x, y, z + 1] from two different ligands and two halide ligands in a distorted tetrahedral geometry (Fig. 2, and Tables 1, 3 and 5). The coordination behaviour of the Hg^{II} atom is similar to that observed in Hg{2,5-bis(3-pyridyl)-1,3,4-oxadiazole}I₂ (Dong et al., 2003), namely a distorted tetrahedral environment consisting of two N-atom donors from two oxadiazole bridging ligands and two coordinated iodide counter-ions. The dihedral angle between the two terminal pyridine ring planes changes from 14.121 (6)° in free ligand (I) to 86.818 (2)° in (II), clearly as a result of coordination to Hg^{II} and interactions with the methanol solvent molecule. Additionally, the planes of the two pyridyl rings are rotated relative to that of the oxadiazole ring, with dihedral angles of 87.324 (2) and 7.169 (2) $^{\circ}$ compared with those given above for (I). The free methanol molecules are fixed in the framework via $O-H \cdots N$ and $O-H \cdots O$ hydrogen bonds involving an oxadiazole N atom and an ether O atom of the ligand (Tables 2, 4 and 6).

In the extended structure of (II), the complexes are joined to form a one-dimensional chain running parallel to the c axis (Fig. 3). The bending of the ligand and its coordination at the Hg^{II} centre result in the chain adopting a helical twist. In our previous study (Dong et al., 2003), 2,5-bis(3-pyridyl)-1,3,4oxadiazole (L6) is also coordinated to an Hg^{II} centre to form a single helical chain. The two pyridyl groups in L6 are not coplanar, although the dihedral angle between them is only 12°, while the two terminal pyridine planes in (I) are nearly coplanar. Compared with the short and rigid bidentate groups in L6, the two terminal pyridylmethyl groups of L rotate freely because of the ether O atoms. The length and the coordinating orientation of the ligand are the primary points of difference between the two helical chains. In $Hg(L6)I_2$, the intrachain Hg $\cdot \cdot$ Hg contact is 8.867 (3) Å and the shortest interpolymer Hg \cdots Hg distance is 5.537 (3) Å, with no bonding interactions observed between the different chains. In (II), the intrachain



Figure 3

The extended structure of a helical chain and the intramolecular hydrogen bonding (dashed lines) in (II).



Figure 4

The two-dimensional sheet in (II), constructed by $\pi - \pi$ stacking interactions (green and purple dashed lines in the electronic version of the paper). Some H atoms and the methanol solvent molecules have been omitted.

Hg...Hg contact is 13.066 (3) Å and, because of interchain π - π interactions (see below), the shortest interpolymer Hg···Hg distance is 4.454 (2) Å.

The helical chains in (II) are arranged side by side along the a axis (Fig. 4), where they interact via interchain $\pi - \pi$ contacts between the oxadiazole rings [centroid-centroid distance 3.328 (2) Å], and between the oxadiazole ring on one chain and a pyridine ring on an adjacent chain [centroid-centroid distance = 3.502(1) Å]. The result is that a novel twodimensional sheet is generated in the ac plane.

In summary, three new compounds with a common helical chain motif have been successfully obtained based on the new 1,3,4-oxadiazole bridging bent organic ligand (I) and HgX_2 (X = Cl, Br or I). The helices assemble through interchain $\pi - \pi$ interactions. This study demonstrates that the combination of divergent organic ligands with different metal nodes represents a very useful synthetic route to metal-organic helicates, and that $\pi - \pi$ interactions play an important role in constructing high-dimensional supramolecular compounds.

Experimental

For the preparation of (I), 2,5-bis(2-hydroxy-5-methylphenyl)-1,3,4oxadiazole (1.03 g, 4.0 mmol), potassium iodide (0.35 g, 2.0 mmol) and potassium carbonate (5.52 g, 40.0 mmol) were combined in dry dimethylformamide (20 ml) with stirring at ambient temperature, after which 4-(chloromethyl)pyridine hydrochloride (1.32 g, 8.0 mmol) was added to the suspension. The mixture was stirred for 18 h at room temperature (monitored by thin-layer chromatography), and then water (100 ml) was added. The precipitate which formed was separated by filtration, washed several times with water, and purified on a silica-gel column using tetrahydrofuran as the eluent to afford (I) as a white crystalline solid (yield 0.95 g, 51%). A solution of (I) (9.28 mg, 0.020 mmol) in CH₂Cl₂ (10 ml) was left for about 2 d at room temperature after which time colourless crystals were obtained (yield 6.01 mg, 65%; m.p. 455–457 K). IR (KBr pellet, ν , cm⁻¹): 3425 (m), 3052 (w), 2920 (w), 1605 (m), 1561 (w), 1515 (s), 1451 (m), 1420 (m), 1386 (m), 1333 (w), 1296 (s), 1268 (s), 1158 (w), 1090 (w), 1061 (m), 993 (w), 887 (w), 812 (m), 786 (w), 757 (w), 683 (w), 610 (w), 549 (w), 488 (w); ¹H NMR (300 MHz, DMSO, 298 K, TMS): δ 8.50 (d, 4H, $-C_5H_4N$, 7.75 (s, 2H, $-C_6H_4$), 7.54 (d, 4H, $-C_5H_4N$), 7.43–7.46 (d, 2H, -C₆H₄), 7.25-7.28 (d, 2H, -C₆H₄), 5.30 (s, 4H, -CH₂), 2.31 (s, 6H, -CH₃). Elemental analysis calculated for C₂₈H₂₄N₄O₃: C 72.40, H 5.21, N 12.06%; found: C 72.11, H 5.08, N 12.00%.

A solution of HgCl₂ (5.42 mg, 0.020 mmol) in CH₃OH (5 ml) was layered on to a solution of (I) (9.28 mg, 0.020 mmol) in CH₂Cl₂ (8 ml). The system was left for about a week at room temperature and colourless crystals of (II) were obtained (yield 9.33 mg, 60%). IR (KBr pellet, ν , cm⁻¹): 3356 (m), 1611 (s), 1535 (s), 1513 (w), 1449 (m), 1425 (w), 1384 (w), 1290 (m), 1268 (s), 1217 (w), 1155 (w), 1062 (m), 1046 (m), 1011 (m), 810 (s), 757 (w), 623 (w), 546 (w).

A solution of HgBr₂ (7.21 mg, 0.020 mmol) in CH₃OH (5 ml) was layered on to a solution of (I) (9.28 mg, 0.020 mmol) in CH₂Cl₂ (8 ml). The system was left for about two weeks at room temperature and colourless crystals of (III) were obtained (yield 7.01 mg, 41%). IR (KBr pellet, ν , cm⁻¹): 3349 (*m*), 1612 (*s*), 1562 (*w*), 1535 (*m*), 1512 (*s*), 1449 (*m*), 1424 (*w*), 1385 (*m*), 1289 (*m*), 1267 (*s*), 1217 (*w*), 1155 (*w*), 1062 (*m*), 1046 (*s*), 1011 (*s*), 890 (*w*), 810 (*w*), 757 (*s*), 624 (*w*), 546 (*w*).

A solution of HgI₂ (5.42 mg, 0.020 mmol) in CH₃OH (5 ml) was layered on to a solution of (I) (9.28 mg, 0.020 mmol) in CH₂Cl₂ (8 ml). The system was left for about 3 d at room temperature and colourless crystals of (IV) were obtained (vield 7.41 mg, 39%). IR (KBr pellet, ν , cm⁻¹): 3442 (*m*), 1613 (*s*), 1536 (*m*), 1512 (*s*), 1450 (*m*), 1426 (m), 1384 (s), 1290 (w), 1268 (m), 1217 (s), 1156 (m), 1062 (w), 1047 (m), 1011 (m), 810 (m), 757 (w), 624 (w), 546 (w).

Compound (I)

Crystal data	
$C_{28}H_{24}N_4O_3$	$V = 2369.4 (14) \text{ Å}^3$
$M_r = 464.51$	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 12.479 (4) Å	$\mu = 0.09 \text{ mm}^{-1}$
b = 11.452 (4) Å	$T = 298 { m K}$
c = 16.776 (6) Å	$0.30 \times 0.22 \times 0.17 \text{ mm}$
$\beta = 98.766 \ (6)^{\circ}$	

Data collection

Bruker SMART CCD area-detector	4420 independent reflections
diffractometer	2887 reflections with $I > 2\sigma(I)$
12163 measured reflections	$R_{\rm int} = 0.032$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.053$ 318 parameters $wR(F^2) = 0.141$ H-atom parameters constrained S = 1.02 $\Delta \rho_{\rm max} = 0.17 \text{ e } \text{\AA}^{-3}$ $\Delta \rho_{\rm min} = -0.17 \text{ e} \text{ Å}^{-3}$ 4420 reflections

Compound (II)

Crystal data

 $[Hg_2Cl_2(C_{28}H_{24}N_4O_3)]$ ·CH₄O $\gamma = 99.318 \ (3)^{\circ}$ $M_r = 768.04$ V = 1450.5 (5) Å³ Triclinic, P1 Z = 2a = 8.8422 (18) ÅMo $K\alpha$ radiation $\mu = 5.53 \text{ mm}^{-1}$ b = 12.927 (3) Å c = 13.066 (3) Å T = 298 K $\alpha = 95.232 \ (3)^{\circ}$ $\beta = 97.758 \ (3)^{\circ}$

Data collection

Bruker SMART CCD area-detector diffractometer Absorption correction: multi-scan (SADABS; Bruker, 2003) $T_{\min} = 0.472, \ T_{\max} = 0.666$

 $0.16 \times 0.14 \times 0.08 \text{ mm}$

7519 measured reflections 5294 independent reflections 4319 reflections with $I > 2\sigma(I)$ $R_{\rm int}=0.036$

metal-organic compounds

Table 1 Selected geometric parameters (Å, °) for (II).

Cl1-Hg1	2.352 (2)	Hg1-N3	2.416 (6)
Cl2-Hg1	2.348 (2)	Hg1-N4 ⁱ	2.441 (6)
Cl2-Hg1-Cl1	149.21 (10)	Cl2-Hg1-N4 ⁱ	105.22 (16)
Cl2-Hg1-N3	97.36 (15)	Cl1-Hg1-N4 ⁱ	98.30 (16)
Cl1-Hg1-N3	102.37 (15)	N3-Hg1-N4 ⁱ	89.80 (19)

Symmetry code: (i) x, y, z + 1.

Table 2

Hydrogen-bond geometry (Å, °) for (II).

$D - H \cdot \cdot \cdot A$	$D-\mathrm{H}$	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdot \cdot \cdot A$
$O4-H4A\cdots N1$	0.82	2.05	2.862 (8)	174
$O4-H4A\cdots O3$	0.82	2.45	2.856 (7)	112

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.052$ 363 parameters $wR(F^2) = 0.129$ H-atom parameters constrained S = 1.00 $\Delta \rho_{\rm max} = 1.71 \ {\rm e} \ {\rm \AA}^{-3}$ $\Delta \rho_{\rm min}$ = -2.51 e Å⁻³ 5294 reflections

Compound (III)

Crystal data

[Hg2Br2(C28H24N4O3)]·CH4O $\gamma = 98.902 \ (2)^{\circ}$ $M_r = 856.96$ V = 1441.5 (3) Å³ Triclinic, $P\overline{1}$ Z = 2a = 8.8247 (12) ÅMo $K\alpha$ radiation b = 12.9673 (18) Å $\mu = 8.15 \text{ mm}^{-1}$ c = 13.0197 (18) Å T = 123 K $\alpha = 95.747 \ (2)^{\circ}$ $0.40 \times 0.28 \times 0.10 \; \mathrm{mm}$ $\beta = 99.101 \ (2)^{\circ}$

Data collection

Bruker SMART CCD area-detector	7538 measured reflections
diffractometer	5274 independent reflections
Absorption correction: multi-scan	4754 reflections with $I > 2\sigma(I)$
(SADABS; Bruker, 2003)	$R_{\rm int} = 0.036$
$T_{\min} = 0.139, T_{\max} = 0.496$	

Table 3

Selected geometric parameters (Å, $^{\circ}$) for (III).

2.4914 (7)	Hg1-N3	2.405 (5)
2.4844 (8)	Hg1-N4 ⁱ	2.415 (5)
80.01 (17)	N2 Hal Drl	104 20 (12)
89.01 (17)	N3-Hg1-Bf1	104.29 (13)
97.87 (14)	N4 ¹ -Hg1-Br1	99.03 (13)
108.12 (13)	Br2-Hg1-Br1	145.07 (3)
	2.4914 (7) 2.4844 (8) 89.01 (17) 97.87 (14) 108.12 (13)	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$

Symmetry code: (i) x, y, z + 1.

Table 4

Hydrogen-bond g	geometry (Å,	$^{\circ}$) for	(III).
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$D - H \cdots A$	$D-\mathrm{H}$	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdots A$
$O4-H4A\cdots N1$	0.84	2.04	2.865 (7)	167
$O4-H4A\cdots O3$	0.84	2.34	2.818 (6)	116

Table 5

Selected geometric parameters (Å, °) for (IV).

I1-Hg1	2.6482 (7)	Hg1-N3	2.427 (6)
I2-Hg1	2.6440 (9)	Hg1-N4 ⁱ	2.429 (6)
$\begin{array}{l} N3-Hg1-N4^{i}\\ N3-Hg1-I2\\ N4^{i}-Hg1-I2 \end{array}$	90.0 (2)	N3-Hg1-I1	105.39 (14)
	98.62 (15)	N4 ⁱ -Hg1-I1	100.77 (14)
	108.08 (14)	I2-Hg1-I1	142.20 (3)

Symmetry code: (i) x, y, z + 1.

Table 6

Hydrogen-bond geometry (Å, $^{\circ}$) for (IV).

$D - H \cdot \cdot \cdot A$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdots A$
$O4-H4A\cdots N1$	0.82	2.08	2.878 (9)	164
$O4-H4A\cdots O3$	0.82	2.36	2.855 (8)	119

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.048$ 363 parameters $wR(F^2) = 0.127$ H-atom parameters constrained S = 1.02 $\Delta \rho_{\rm max} = 2.73 \ {\rm e} \ {\rm \AA}^{-3}$ $\Delta \rho_{\rm min} = -3.43 \text{ e} \text{ Å}^{-3}$ 5274 reflections

Compound (IV)

Crystal data

$[Hg_2I_2(C_{28}H_{24}N_4O_3)] \cdot CH_4O$	$\gamma = 97.271 \ (4)^{\circ}$
$M_r = 950.94$	V = 1525.4 (7) Å ³
Triclinic, P1	Z = 2
a = 9.046 (3) Å	Mo $K\alpha$ radiation
b = 13.153 (4) Å	$\mu = 7.11 \text{ mm}^{-1}$
c = 13.213 (4) Å	$T = 298 { m K}$
$\alpha = 95.975 \ (4)^{\circ}$	$0.21 \times 0.10 \times 0.04 \text{ mm}$
$\beta = 99.503 \ (4)^{\circ}$	

Data collection

Bruker SMART CCD area-detector	8032 measured reflections
diffractometer	5543 independent reflections
Absorption correction: multi-scan	4328 reflections with $I > 2\sigma(I)$
(SADABS; Bruker, 2003)	$R_{\rm int} = 0.036$
$T_{\min} = 0.317, \ T_{\max} = 0.764$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$	363 parameters
$wR(F^2) = 0.107$	H-atom parameters constrained
S = 1.01	$\Delta \rho_{\rm max} = 1.29 \text{ e} \text{ Å}^{-3}$
5543 reflections	$\Delta \rho_{\rm min} = -1.03 \text{ e } \text{\AA}^{-3}$

H atoms attached to anisotropically refined atoms were placed in geometrically idealized positions and included as riding atoms with the following constraints: for (I), (II) and (IV), C-H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2 U_{\rm eq}({\rm C})$ (aromatic), C-H = 0.96 Å and $U_{\rm iso}({\rm H}) =$ $1.5U_{eq}(C)$ (methyl), and C-H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ (methylene). For (II) and (IV), O-H = 0.82 Å and $U_{iso}(H) =$ 1.5 $U_{eq}(O)$ (methanol). For (III), C-H = 0.95 Å and $U_{iso}(H)$ = $1.2U_{eq}(C)$ (aromatic), C-H = 0.98 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ (methyl), C-H = 0.99 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ (methylene), and O-H = 0.84 Å and $U_{iso}(H) = 1.5U_{eq}(O)$ (methanol). In (II), the maximum and minimum residual electron-density peaks are 1.24 and 1.14 Å, respectively, from atom Hg1. In (III), these peaks are 0.88 and 0.86 Å, respectively, from atom Hg1, and in (IV), they are 1.00 and 1.64 Å, respectively, from atom Hg1.

metal-organic compounds

For all compounds, data collection: *SMART* (Bruker, 2003); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SQ3288). Services for accessing these data are described at the back of the journal.

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