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Bis(1,2-di-4-pyridylethene)bis(5-hydroxyisophthalato)zirconium(II) monohydrate

Ya-Juan Zhao, Ruo-Wang Liu and Xin-Hua Li*

School of Chemistry and Materials Science, Wenzhou Normal College, Zhejiang, Wenzhou 325027, People's Republic of China

Correspondence e-mail: lixinhua01@126.com

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In the title compound, $[Zr(C_8H_5O_5)_2(C_{12}H_{10}N_2)_2]\cdot H_2O$, each Zr atom is surrounded by four carboxylate O atoms of two 5-hydroxyisophthalate anions and two N atoms of two 1,2-di-4-pyridylethene ligands, the 5-hydroxyisophthalate anion functioning in a chelating coordination mode. The Zr atom is six-coordinate and the geometry is strongly distorted octahedral. The Zr cation and the water molecule lie on a crystallographic twofold axis.

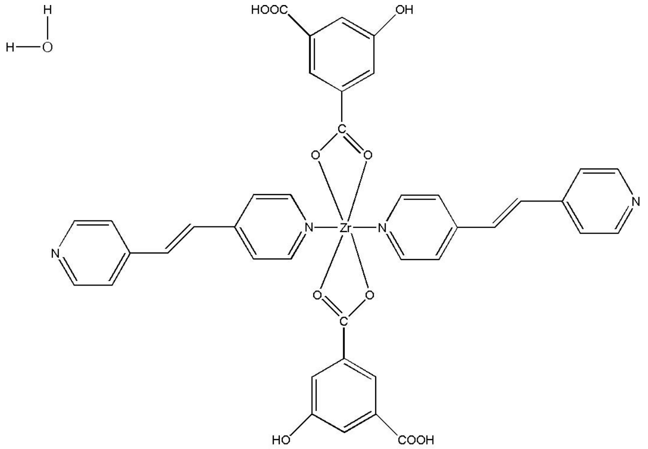
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.064
wR factor = 0.194
Data-to-parameter ratio = 12.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Much interest at present is focused on the construction of coordination polymers (Carlucci *et al.*, 1994; Munakata *et al.*, 1999; Hirsch *et al.*, 1997; Hoskins & Robson, 1990), and much of this interest has involved linear pyridyl-donor ligands. These include pyrazine (Carlucci *et al.*, 1995), 4,4′-bipyridine (Yaghi & Li, 1996) and longer bridges (Soma & Iwamoto, 1997). Bipyridine has been used extensively in the past (Huang & Xiong, 1997); however, few coordination polymers are known for other ligands (Batten *et al.*, 1999). Against this background, we report here the structure of the title compound, (I), to outline further studies on coordination polymers constructed through the interaction of metal ions with pyridyl-donor ligands with two coordinating sites.



(I)

The title compound consists of the bis(1,2-di-4-pyridylethene)bis(5-hydroxyisophthalato)zirconium(II) complex and uncoordinated water molecules (Fig. 1) linked by hydrogen bonds into a network structure (Fig. 2 and Table 2). The Zr atom and the water molecule lie on a crystallographic twofold axis. The Zr atom is six-coordinated by four carboxylate O atoms of two 5-hydroxyisophthalate anions and two N atoms of two 1,2-di-4-pyridylethene ligands. The geometry around the Zr atom is strongly distorted octahedral, and the Zr atom

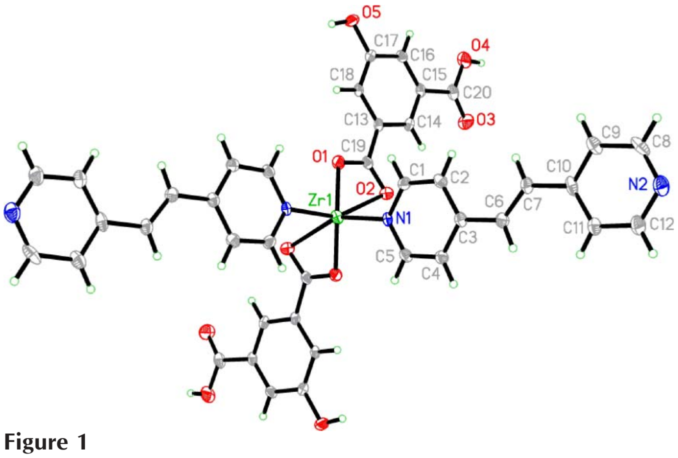


Figure 1
ORTEPII plot (Johnson, 1976) of the title compound, showing 50% probability displacement ellipsoids. Only the contents of the asymmetric unit are labeled. Unlabeled atoms are related to labeled atoms by ($2 - x$, y, $\frac{3}{2} - z$). The water molecule has been omitted for clarity.

is displaced from the center of the octahedron along the twofold axis. Such a distortion is usually found for a d^0 transition metal in an octahedral coordination (Kunz & Brown, 1995). The 1,2-di-4-pyridylethene ligand acts as a monodentate ligand and the 5-hydroxyisophthalate anion functions in a chelating coordination mode.

Experimental

Zirconium carbonate dihydrate (0.4 g, 2 mmol) was dissolved in a dilute hydrochloric acid solution (10 ml, pH = 3) and the solution was mixed with a dimethylformamide solution (10 ml) of 1,2-di-4-pyridylethene (0.4 g, 2 mmol), 5-hydroxyisophthalic acid (0.4 g, 2 mmol) and 2,2′-dithiosalicylic acid (0.6 g, 2 mmol) at 298 K. The reaction mixture was filtered and colorless prism-shaped crystals separated from the solution after about three months.

Crystal data

$[Zr(C_8H_5O_5)_2(C_{12}H_{10}N_2)_2]\cdot H_2O$
$M_r = 835.92$
Monoclinic, $C2/c$
$a = 34.037$ (3) Å
$b = 8.3818$ (7) Å
$c = 13.0137$ (11) Å
$\beta = 103.502$ (1)°
$V = 3610.1$ (5) Å^3
$Z = 4$
$D_x = 1.538$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 3197 reflections
$\theta = 1.2$–25.1°
$\mu = 0.38$ mm^{-1}
$T = 298$ (2) K
Prism, colorless
0.33 × 0.30 × 0.07 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.88$, $T_{max} = 0.97$
9198 measured reflections
3197 independent reflections
2859 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 25.1°$
$h = -31 \rightarrow 40$
$k = -8 \rightarrow 10$
$l = -15 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.194$
$S = 1.12$
3197 reflections
259 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1021P)^2 + 20.1938P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.83$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

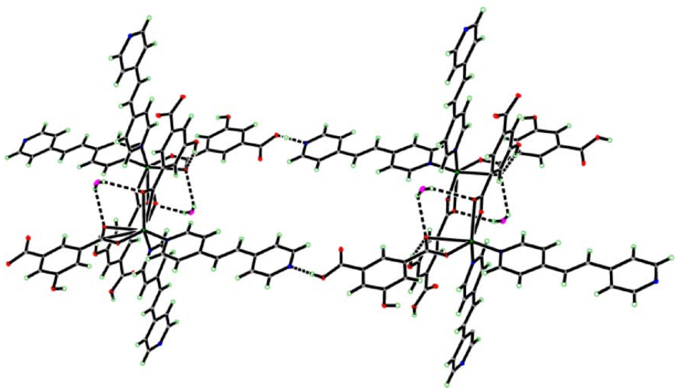


Figure 2
The three-dimensional network formed by hydrogen-bonding interactions (shown as dashed lines) in the title compound.

Table 1
Selected geometric parameters (Å, °).

Zr1—O1	1.975 (3)	Zr1—O2	2.623 (4)
Zr1—N1	2.066 (3)		
O1—Zr1—O1^{i}	142.6 (2)	N1—Zr1—O2^{i}	153.06 (13)
O1—Zr1—N1^{i}	98.12 (14)	O1—Zr1—O2	54.99 (12)
O1—Zr1—N1	104.97 (14)	N1—Zr1—O2	87.38 (13)
N1^{i}—Zr1—N1	103.0 (2)	O2^{i}—Zr1—O2	94.44 (16)
O1—Zr1—O2^{i}	97.92 (13)		

Symmetry code: (i) $2 - x, y, \frac{3}{2} - z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6A···O2^{ii}	0.82 (14)	2.50 (15)	3.024 (8)	122 (15)
O5—H5···O2^{iii}	0.82	1.95	2.747 (5)	164
O4—H4A···N2^{iv}	0.82	1.83	2.644 (6)	175

Symmetry codes: (ii) $1 - x, y - 1, \frac{3}{2} - z$; (iii) $x, 2 - y, z - \frac{1}{2}$; (iv) $\frac{3}{2} - x, \frac{3}{2} + y, \frac{3}{2} - z$.

The water H atoms were refined subject to the restraint O—H = 0.82 (5) Å. The other H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of 0.82 (O—H) and 0.93 Å (C—H), with $U_{iso} = 1.2U_{eq}$(O,C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 2002) and *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXTL* (Bruker, 2002).

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