metal-organic papers

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Key indicators

Single-crystal X-ray study
$T = 292$ K
Mean σ(C–C) = 0.006 Å
R factor = 0.045
wR factor = 0.106
Data-to-parameter ratio = 14.2

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

catena-Poly[[[pyrazino[2,3-f][1,10]phenanthroline]zinc(II)]-μ4-fumarato-μ2-fumarato]

In the title compound, [Zn(C$_4$H$_2$O$_4$)(C$_{14}$H$_8$N$_4$)]$_n$, the Zn^{II} atom is five-coordinate and exhibits a distorted trigonal–bipyramidal coordination. The Zn^{II} atoms are further bridged by fumarate ligands, forming a two-dimensional network parallel to the *ab* plane.

Comment

The use of unsaturated organic acids such as fumarate as flexible spacers in the syntheses of coordination polymers has aroused enormous interest in recent years because of their versatile coordination modes and varieties of structural conformations (Zhu *et al.*, 2006). On the other hand, the 1,10-phenanthroline (phen) ligand has been widely used in the construction of metal-organic complexes (Chen & Liu, 2002), while an important derivative of phen, namely pyrazino[2,3-f][1,10]phenanthroline (Pyphen), was recently used to synthesize coordination polymers (Che *et al.*, 2006). We selected fumaric acid (H$_2$fum) as a linker and Pyphen as a secondary chelating ligand, generating a new Zn^{II} coordination polymer, [Zn(fum)(Pyphen)]$_n$, (I), which is reported here.

(I)
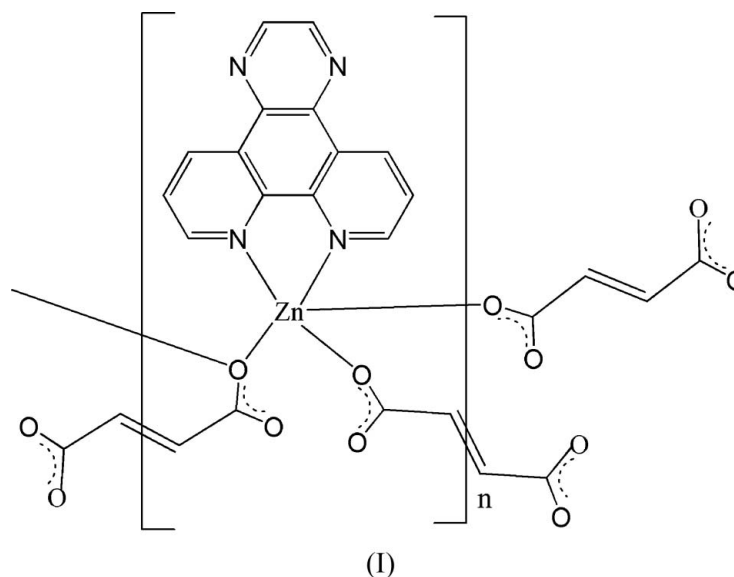
Selected bond lengths and angles for (I) are given in Table 1. In compound (I), each Zn^{II} atom is five-coordinate and exhibits a distorted trigonal–bipyramidal coordination (Fig. 1). Two carboxylate O atoms (O1 and O3) from two fum ligands and one N atom (N1) from the Pyphen ligand form the equatorial plane, while atoms N2 and O1($1 - x, 1 - y, 1 - z$) of the Pyphen and fum ligands, respectively, occupy the axial positions.
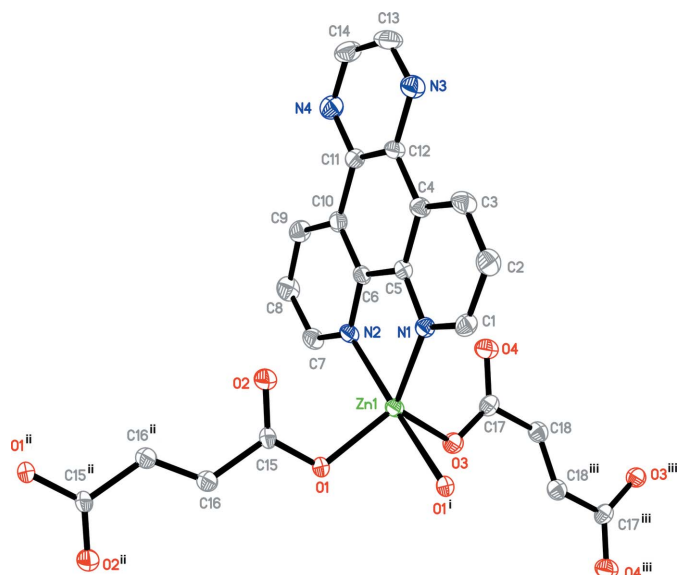
Figure 1
The asymmetric unit of (I), together with further atoms to complete the Zn^{II} coordination and the fumarate ligands. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry codes: (i) $1 - x$, $1 - y$, $1 - z$; (ii) $1 - x$, $-y$, $1 - z$; (iii) $2 - x$, $2 - y$, $1 - z$.]
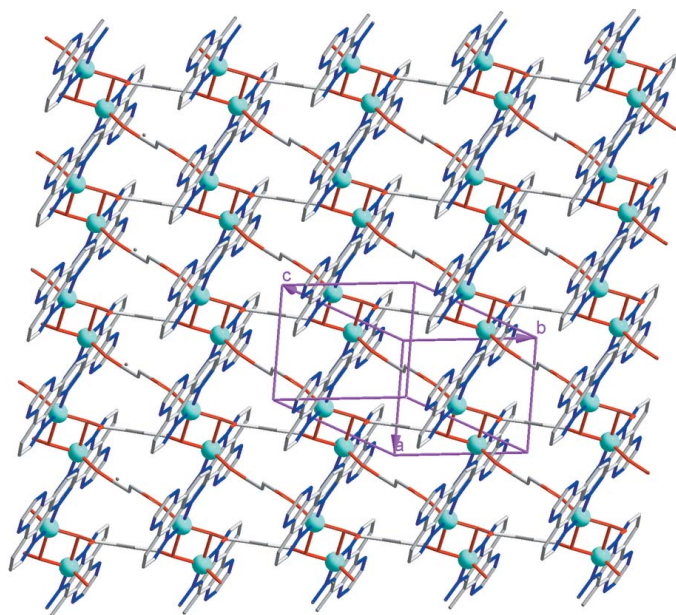
Figure 2
View of the layered structure of (I). H atoms have been omitted for clarity.

The Zn^{II} ions are bridged by fum ligands, generating a two-dimensional network with (4,4)-grids parallel to the ab plane (Fig. 2). These layers are decorated with Pyphen ligands on both sides. Each corner of the (4,4)-grid is occupied by a binuclear Zn^{II} subunit. No π–π interactions involving the Pyphen ring system is observed, but the structure contains weak C—H⋯O interactions (Table 2).

Experimental

The Pyphen ligand was synthesized according to the literature method of Dickeson & Summers (1970). A methanolic solution (16 ml) of Pyphen (0.5 mmol) was added slowly to an aqueous solution (20 ml) of ZnCl$_2$·H$_2$O (0.5 mmol) and H$_2$fum (1 mmol) with stirring. The resulting solution was filtered and the filtrate was allowed to stand in air at room temperature for several days, yielding colourless crystals of (I) (28% yield based on Zn).

Crystal data

[Zn(C₄H₂O₄)(C₁₄H₈N₄)]	$V = 797.8$ (3) Å^3
$M_r = 411.67$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.714$ Mg m^{-3}
$a = 6.8613$ (14) Å	Mo $K\alpha$ radiation
$b = 8.3961$ (17) Å	$\mu = 1.58$ mm^{-1}
$c = 14.610$ (3) Å	$T = 292$ (2) K
$\alpha = 105.08$ (3)°	Block, colourless
$\beta = 98.70$ (3)°	$0.22 \times 0.19 \times 0.18$ mm
$\gamma = 94.15$ (3)°	

Data collection

Rigaku R-AXIS RAPID diffractometer	7523 measured reflections
ω scans	3472 independent reflections
Absorption correction: multi-scan ($ABSCOR$; Higashi, 1995)	2651 reflections with $I > 2\sigma(I)$
$T_{min} = 0.700$, $T_{max} = 0.755$	$R_{int} = 0.040$
	$\theta_{max} = 27.3°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.045$	$+ 1.1513P]$
$wR(F^2) = 0.106$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} = 0.001$
3472 reflections	$\Delta\rho_{max} = 0.46$ e Å^{-3}
244 parameters	$\Delta\rho_{min} = -0.48$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Zn1—O3	1.996 (3)	Zn1—N2	2.150 (3)
Zn1—O1	2.019 (2)	Zn1—O1^i	2.425 (3)
Zn1—N1	2.107 (3)		
O3—Zn1—O1	113.16 (11)	N1—Zn1—N2	79.02 (11)
O3—Zn1—N1	118.42 (12)	O3—Zn1—O1^i	84.24 (10)
O1—Zn1—N1	125.98 (12)	O1—Zn1—O1^i	77.65 (10)
O3—Zn1—N2	105.43 (12)	N1—Zn1—O1^i	92.25 (10)
O1—Zn1—N2	102.26 (11)	N2—Zn1—O1^i	169.21 (10)

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

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C7—H7⋯O3^{ii}	0.93	2.41	3.337 (5)	174
C13—H13⋯O4^{iii}	0.93	2.37	3.204 (6)	149
C14—H14⋯O2^{iv}	0.93	2.52	3.344 (5)	148

Symmetry codes: (ii) $-x + 2$, $-y + 1$, $-z + 1$; (iii) $-x + 1$, $-y + 1$, $-z$; (iv) $-x + 1$, $-y$, $-z$.

All H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

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Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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