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catena-Poly[μ_2-oxalato-$\kappa^4O,O':O'',O'''$-bis(pyridine-κN)zinc(II)]

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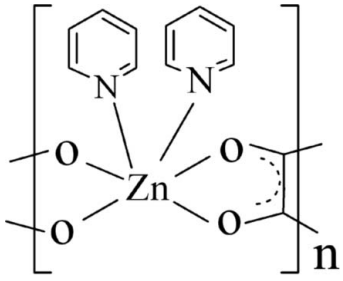
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.030; wR factor = 0.080; data-to-parameter ratio = 17.3.

The title compound, $[Zn(C_2O_4)(C_5H_5N)_2]_n$, was synthesized by the oxidation of pentaerythritol under solvothermal conditions. The backbone of the compound is formed from Zn^{II} oxalate, with two pyridine ligands coordinated to each Zn^{II} ion, giving it hexacoordination in a slightly distorted octahedral environment.

Related literature

For related literature, see: Chui *et al.* (1999); Evans & Lin (2001); Ghosh *et al.* (2004); Kahn & Martinez (1998); Kiang *et al.* (1999); Lin *et al.* (1999).



Experimental

Crystal data

$[Zn(C_2O_4)(C_5H_5N)_2]$
M_r = 311.59
Monoclinic, $P2_1/n$
a = 9.4780 (9) Å
b = 9.2118 (8) Å
c = 15.0863 (13) Å
β = 94.402 (4)°
V = 1313.3 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.88 mm^{-1}
T = 293 (2) K
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.602, T_{max} = 0.705
15885 measured reflections
2981 independent reflections
2030 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.081
S = 0.82
2981 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O1	2.0935 (15)	Zn1—O4	2.1365 (16)
Zn1—O2	2.0987 (15)	Zn1—N2	2.1485 (19)
Zn1—O3	2.1269 (16)	Zn1—N1	2.161 (2)
O1—Zn1—O2	172.80 (6)	O1—Zn1—O4	78.81 (6)
O1—Zn1—O3	97.56 (6)	O2—Zn1—O4	94.69 (6)
O2—Zn1—O3	79.08 (6)	O3—Zn1—O4	89.14 (7)

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997*b*).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2024).

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supplementary materials

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catena-Poly[μ_2-oxalato-$\kappa^4O,O':O'',O'''$-bis(pyridine-κN)zinc(II)]

H.-G. Yao, M. Ji, L.-J. Zou and Y.-L. An

Comment

Synthesis of metal organic framework (MOF) structures by the modular approach is an area of intense research activity as potential zeolitic, optoelectronic, magnetic, and conducting materials(Chui *et al.*, 1999; Kiang *et al.*, 1999; Kahn & Martinez, 1998; Lin *et al.*, 1999). While most of these products have been generated utilizing hydro(solvo)thermal techniques, it is often not possible to predict the structures of them with confidence (Evans & Lin, 2001; Ghosh *et al.*, 2004). In this paper, we report our unexpected discovery of the synthesis of a new zinc oxalate coordination polymers by facile oxidation of pentaerythritol acid to oxalic acid under hydro(solvo)thermal conditions.

The structure of the compound consists of infinite one-dimensional zigzag chains where each metal ion is coordinated to two oxalate groups and two pyridine molecules showing hexacoordination with O_4N_2 donor set (Fig. 1). Coordination geometry around each metal center can be described as slightly distorted octahedral. The two pyridine molecules are similarly oriented with respect to the Zn-oxalate backbone. These infinite zigzag chains pack in the lattice through interdigitization involving the pyridine molecules (Fig. 2)·The Zn—O distances range from 2.0935 (15) Å to 2.1365 (16) Å, while the O—Zn—O angles between 78.81 (6)° and 172.80 (6) (Table 1).

Experimental

The compound was prepared by a solvothermal reaction of pentaerythritol (0.031 g), $Zn(NO_3)_2{\cdot}6H_2O$ (0.027 g), using a solvent of pyridine (0.475 g). The mixture was sealed in a Pyrex glass tube with *ca* 10% filling, placed into a stainless-autoclave, and heated at 393 K for 6 days. After cooling naturally to ambient temperature, the products were washed with ethanol, and the yellow block crystals were obtained.

Refinement

H atoms were positioned geometrically with C—H = 0.93 Å and allowed to ride during subsequent refinement with U_{iso}(H) = 1.2U_{eq}(C).

Figures

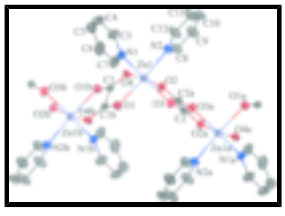

Fig. 1. View of the molecular structure of (I) with the atom-labelling scheme and displacement ellipsoids drawn at the 30% probability level. Symmetry code:(i) 2 − *x*, −1 − *y*,-*z* + 1; (ii) −*x* + 1, −*y* − 1, −*z* + 1. H atoms have been omitted.

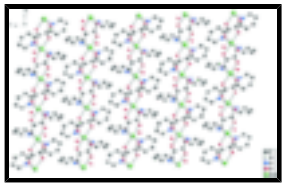

Fig. 2. Packing of the zigzag chains through interchain interdigitization of pyridine molecules in the compound. H atoms have been omitted.

catena-poly[μ$_2$-oxalato-κ^4*O*,*O*':*O*'',*O*'''-bis(pyridine-κ*N*)zinc(II)]

Crystal data

$[Zn(C_2O_4)(C_5H_5N)_2]$	F_{000} = 632
M_r = 311.59	D_x = 1.576 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 9.4780 (9) Å	Cell parameters from 2981 reflections
b = 9.2118 (8) Å	θ = 2.5–26.3º
c = 15.0863 (13) Å	μ = 1.88 mm^{-1}
β = 94.402 (4)º	T = 293 (2) K
V = 1313.3 (2) Å^3	Block, yellow
Z = 4	0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEX CCD diffractometer	2981 independent reflections
Radiation source: fine-focus sealed tube	2030 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.067
T = 293(2) K	θ_{max} = 27.4º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	h = −12→12
T_{min} = 0.602, T_{max} = 0.705	k = −11→11
15885 measured reflections	l = −19→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.030	H-atom parameters constrained
$wR(F^2)$ = 0.081	$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 0.8316P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.82	$(\Delta/\sigma)_{max}$ = 0.001
2981 reflections	$\Delta\rho_{max}$ = 0.53 e Å^{-3}
172 parameters	$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.75084 (3)	−0.34984 (3)	0.504672 (15)	0.02915 (10)
O1	0.56734 (16)	−0.36565 (17)	0.57297 (10)	0.0330 (4)
O2	0.92978 (16)	−0.36105 (18)	0.43119 (9)	0.0335 (4)
O3	0.86912 (16)	−0.51562 (18)	0.57471 (10)	0.0359 (4)
O4	0.63625 (16)	−0.51363 (18)	0.42912 (10)	0.0351 (4)
N1	0.6728 (2)	−0.1855 (2)	0.41113 (12)	0.0361 (5)
N2	0.8342 (2)	−0.1952 (2)	0.60162 (12)	0.0340 (5)
C1	0.4796 (2)	−0.4570 (2)	0.54165 (13)	0.0267 (5)
C2	1.0183 (2)	−0.4548 (3)	0.45843 (12)	0.0273 (5)
C3	0.6379 (3)	−0.0519 (3)	0.43339 (17)	0.0508 (7)
H3A	0.6433	−0.0276	0.4934	0.061*
C4	0.5940 (4)	0.0530 (4)	0.37219 (19)	0.0663 (9)
H4A	0.5703	0.1456	0.3907	0.080*
C5	0.5860 (3)	0.0183 (4)	0.28352 (19)	0.0618 (9)
H5A	0.5558	0.0865	0.2407	0.074*
C6	0.6231 (4)	−0.1172 (4)	0.25962 (18)	0.0606 (9)
H6A	0.6201	−0.1430	0.1999	0.073*
C7	0.6655 (3)	−0.2167 (3)	0.32437 (16)	0.0481 (7)
H7A	0.6900	−0.3097	0.3070	0.058*
C8	0.9477 (3)	−0.1161 (3)	0.58687 (16)	0.0474 (7)
H8A	0.9903	−0.1306	0.5340	0.057*
C9	1.0050 (3)	−0.0147 (3)	0.64543 (18)	0.0575 (8)
H9A	1.0835	0.0391	0.6317	0.069*
C10	0.9457 (3)	0.0066 (3)	0.72456 (17)	0.0512 (7)
H10A	0.9831	0.0744	0.7656	0.061*
C11	0.8304 (3)	−0.0743 (4)	0.74121 (17)	0.0556 (8)
H11A	0.7875	−0.0626	0.7942	0.067*
C12	0.7781 (3)	−0.1733 (3)	0.67895 (16)	0.0473 (7)
H12A	0.6995	−0.2279	0.6914	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.02419 (15)	0.03332 (17)	0.03018 (15)	−0.00085 (13)	0.00355 (10)	−0.00017 (12)
O1	0.0294 (9)	0.0360 (10)	0.0342 (8)	−0.0041 (8)	0.0056 (6)	−0.0073 (7)
O2	0.0297 (9)	0.0397 (10)	0.0317 (7)	0.0054 (8)	0.0062 (6)	0.0067 (7)
O3	0.0296 (9)	0.0446 (11)	0.0347 (8)	0.0057 (8)	0.0107 (7)	0.0074 (7)
O4	0.0274 (9)	0.0423 (10)	0.0365 (8)	−0.0053 (8)	0.0089 (7)	−0.0072 (7)
N1	0.0306 (11)	0.0414 (13)	0.0364 (10)	0.0021 (10)	0.0040 (8)	0.0064 (9)
N2	0.0320 (11)	0.0376 (12)	0.0323 (9)	−0.0029 (9)	0.0015 (8)	−0.0024 (9)
C1	0.0236 (12)	0.0290 (13)	0.0274 (10)	0.0011 (10)	0.0005 (9)	0.0015 (9)
C2	0.0258 (12)	0.0314 (14)	0.0245 (10)	−0.0021 (11)	0.0008 (9)	−0.0026 (9)
C3	0.0587 (19)	0.0557 (19)	0.0393 (13)	0.0220 (16)	0.0127 (12)	0.0041 (13)
C4	0.081 (2)	0.056 (2)	0.0629 (18)	0.0327 (19)	0.0142 (17)	0.0092 (16)
C5	0.066 (2)	0.069 (2)	0.0498 (16)	0.0170 (18)	0.0022 (14)	0.0221 (16)
C6	0.075 (2)	0.066 (2)	0.0380 (14)	−0.0004 (18)	−0.0093 (14)	0.0059 (14)
C7	0.0603 (18)	0.0448 (17)	0.0378 (13)	−0.0016 (15)	−0.0060 (12)	−0.0006 (12)
C8	0.0480 (17)	0.0596 (19)	0.0356 (12)	−0.0180 (14)	0.0105 (11)	−0.0063 (12)
C9	0.0552 (19)	0.066 (2)	0.0514 (16)	−0.0291 (16)	0.0068 (13)	−0.0085 (15)
C10	0.0556 (18)	0.0557 (19)	0.0414 (14)	−0.0129 (15)	−0.0027 (12)	−0.0137 (13)
C11	0.0555 (19)	0.072 (2)	0.0409 (14)	−0.0116 (17)	0.0122 (13)	−0.0204 (14)
C12	0.0402 (15)	0.063 (2)	0.0397 (13)	−0.0138 (14)	0.0107 (11)	−0.0091 (13)

Geometric parameters (Å, °)

Zn1—O1	2.0935 (15)	C12—C11	1.374 (4)
Zn1—O2	2.0987 (15)	C7—C6	1.377 (4)
Zn1—O3	2.1269 (16)	C10—C11	1.362 (4)
Zn1—O4	2.1365 (16)	C10—C9	1.372 (4)
Zn1—N2	2.1485 (19)	C8—C9	1.369 (4)
Zn1—N1	2.161 (2)	C5—C6	1.354 (4)
O2—C2	1.251 (3)	C5—C4	1.372 (4)
O1—C1	1.250 (3)	C4—C3	1.378 (4)
O4—C1^{i}	1.244 (3)	C3—H3A	0.930
O3—C2^{ii}	1.243 (3)	C4—H4A	0.930
N2—C12	1.334 (3)	C5—H5A	0.930
N2—C8	1.332 (3)	C6—H6A	0.930
C2—O3^{ii}	1.243 (3)	C7—H7A	0.930
C2—C2^{ii}	1.566 (4)	C8—H8A	0.930
C1—O4^{i}	1.244 (3)	C9—H9A	0.930
C1—C1^{i}	1.559 (4)	C10—H10A	0.930
N1—C3	1.324 (3)	C11—H11A	0.930
N1—C7	1.337 (3)	C12—H12A	0.930
O1—Zn1—O2	172.80 (6)	N2—C12—C11	123.6 (3)
O1—Zn1—O3	97.56 (6)	N1—C7—C6	122.8 (3)
O2—Zn1—O3	79.08 (6)	C11—C10—C9	118.0 (3)

O1—Zn1—O4	78.81 (6)	N2—C8—C9	123.4 (2)
O2—Zn1—O4	94.69 (6)	C10—C11—C12	119.3 (2)
O3—Zn1—O4	89.14 (7)	C6—C5—C4	118.6 (3)
O1—Zn1—N2	89.26 (6)	C5—C6—C7	119.5 (3)
O2—Zn1—N2	97.02 (7)	C8—C9—C10	119.4 (3)
O3—Zn1—N2	89.18 (7)	C3—C4—C5	118.8 (3)
O4—Zn1—N2	167.63 (6)	N1—C3—C4	123.4 (2)
O1—Zn1—N1	96.57 (7)	H3A—C3—N1	118.67
O2—Zn1—N1	86.54 (7)	H3A—C3—C4	118.70
O3—Zn1—N1	165.55 (7)	H4A—C4—C3	120.25
O4—Zn1—N1	90.68 (7)	H4A—C4—C5	120.22
N2—Zn1—N1	93.98 (8)	H5A—C5—C4	120.69
C2—O2—Zn1	114.15 (13)	H5A—C5—C6	120.71
C1—O1—Zn1	114.47 (13)	H6A—C6—C5	120.35
C1^{i}—O4—Zn1	113.00 (13)	H6A—C6—C7	120.35
C2^{ii}—O3—Zn1	113.18 (14)	H7A—C7—C6	118.22
C12—N2—C8	116.4 (2)	H7A—C7—N1	118.28
C12—N2—Zn1	123.04 (15)	H8A—C8—N2	118.38
C8—N2—Zn1	120.56 (16)	H8A—C8—C9	118.39
O3^{ii}—C2—O2	126.43 (19)	H9A—C9—C8	120.51
O3^{ii}—C2—C2^{ii}	117.1 (2)	H9A—C9—C10	120.52
O2—C2—C2^{ii}	116.5 (2)	H10A—C10—C9	120.59
O4^{i}—C1—O1	126.28 (19)	H10A—C10—C11	120.64
O4^{i}—C1—C1^{i}	117.1 (2)	H11A—C11—C10	120.53
O1—C1—C1^{i}	116.6 (2)	H11A—C11—C12	120.57
C3—N1—C7	117.0 (2)	H12A—C12—C11	118.34
C3—N1—Zn1	124.40 (16)	H12A—C12—N2	118.41
C7—N1—Zn1	118.55 (18)		

Symmetry codes: (i) −*x*+1, −*y*−1, −*z*+1; (ii) −*x*+2, −*y*−1, −*z*+1.

Fig. 1

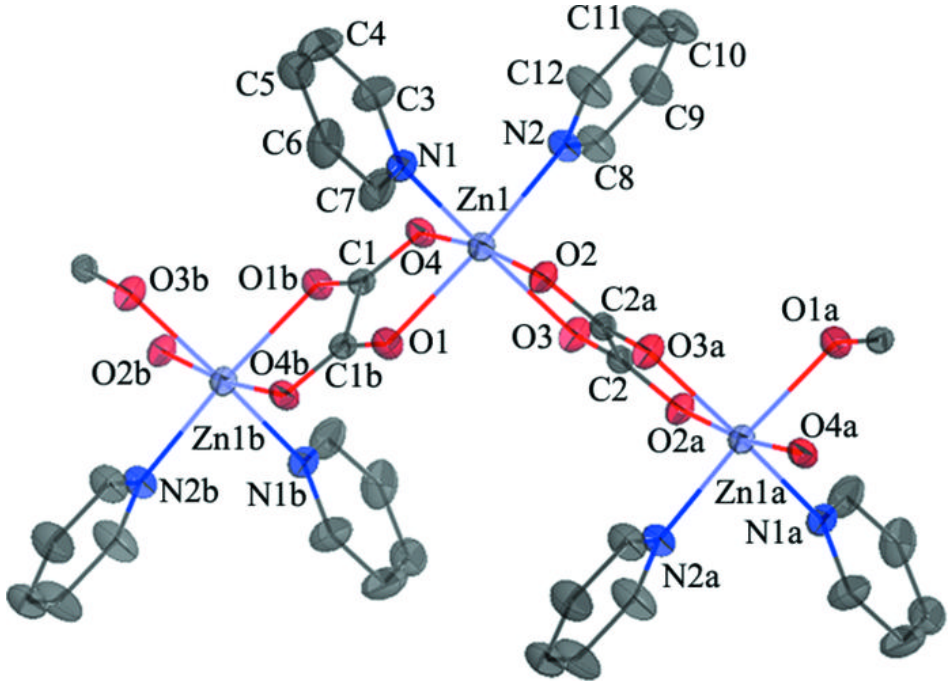
C4
C5
C3
C6
N1
C7
Zn1
C11
C10
C12
C9
N2
C8
O3b
O1b
C1
O4
O2
C2a
O1a
O2b
O4b
C1b
O1
O3
O3a
C2
Zn1b
O2a
O4a
N2b
N1b
Zn1a
N1a
N2a

Fig. 2