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Crystal structure of poly[bis(μ_2-5-hydroxynicotinato-κ^2N:O^3)zinc]

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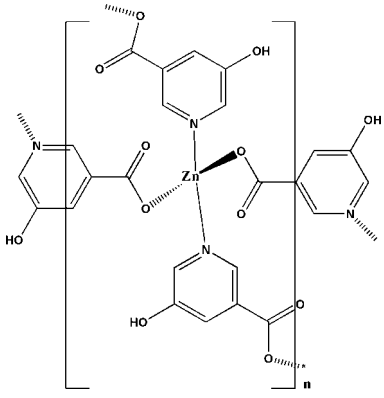
The title coordination polymer, $[Zn(C_6H_4NO_3)_2]_n$, was prepared under hydrothermal conditions by the reaction of zinc nitrate with 5-hydroxynicotinic acid in the presence of malonic acid. In the structure, the Zn^{II} ion is coordinated by two carboxylate O atoms and two pyridine N atoms of four 5-hydroxynicotinate ligands in a distorted tetrahedral coordination environment. The μ_2-bridging mode of each anion leads to the formation of a three-dimensional framework structure. Intermolecular hydrogen bonds between the hydroxy groups of one anion and the non-coordinating carboxylate O atoms of neighbouring anions consolidate the crystal packing.

Keywords: crystal structure; zinc coordination polymer; 5-hydroxynicotinate ligand; hydrogen bonding.

CCDC reference: 1042412

1. Related literature

For transition metal complexes with 5-hydroxynicotinate ligands, see: Jiang & Feng (2008); Zhang *et al.* (2011); Yang *et al.* (2010). For corresponding rare earth metal complexes, see: Zhang *et al.* (2012); Mi *et al.* (2012); Xu *et al.* (2013).



2. Experimental

2.1. Crystal data

$[Zn(C_6H_4NO_3)_2]$
M_r = 341.57
Monoclinic, $P2_1/n$
a = 9.4299 (6) Å
b = 10.5453 (7) Å
c = 12.6914 (8) Å
β = 104.640 (7)°
V = 1221.07 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.04 mm^{-1}
T = 150 K
0.41 × 0.37 × 0.17 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.488, T_{max} = 0.723
7054 measured reflections
2891 independent reflections
2397 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.075
S = 1.07
3345 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O5^{i}	0.82	1.88	2.697 (2)	175
O6—H6A···O2^{ii}	0.82	1.83	2.651 (3)	174

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5106).

References

Brandenburg, K. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2007). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc. Madison, Wisconsin, USA.
Jiang, M.-X. & Feng, Y.-L. (2008). *Acta Cryst.* E**64**, m1517.
Mi, J.-L., Huang, J. & Chen, H.-J. (2012). *Acta Cryst.* E**68**, m1146–m1147.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Xu, S.-S., Mi, J.-L. & Chen, H.-J. (2013). *Acta Cryst.* E**69**, m294–m295.
Yang, J., Chen, H. J. & Tsz, H. L. (2010). *Inorg. Chem. Commun.* **10**, 1016–1019.
Zhang, J., Chen, H.-J. & Huang, J. (2011). *Chin. J. Struct. Chem.* **30**, 1069–1073.
Zhang, J., Huang, J., Yang, J. & Chen, H.-J. (2012). *Inorg. Chem. Commun.* **17**, 163–168.

supporting information

Acta Cryst. (2015). E**71**, m23 [doi:10.1107/S2056989015000249]

Crystal structure of poly[bis(μ_2-5-hydroxynicotinato-κ^2N:O^3)zinc]

Wen-Bing Wang, Shan-Shan Xu and Hong-Ji Chen

S1. Experimental

A mixture of zinc nitrate, 5-hydroxynicotinic acid, malonic acid and water in a mole ratio of *ca* 1:2:1:550 was added to a 25 ml Teflon-lined cup, and the pH value of the mixture was adjusted to 6.5 by 5%$_{wt}$ ammonia/water at room temperature. The Teflon-lined cup was sealed in a stainless steel vessel and heated to 443 K, kept at that temperature for 3 days, and then slowly cooled to room temperature at a rate of 5 K per hour. Yellow block-like crystals of the title compound were obtained. The yield was about 55%. Elemental anal. calc. for $C_{12}H_8N_2O_6Zn$ (341.57): C 28.60, H 2.79, N, 3.28. Found: C 28.65, H 2.81, N, 3.13. IR (cm^{-1}, KBr): 3454(*s*), 3104(*m*), 1856(w), 1632(*s*), 1586(*s*), 1487(*m*), 1432(*m*), 1400(*s*), 1302(w), 1279(*s*), 1239(*m*), 1158(w), 1119(w), 1026(*s*), 968(w), 936(*s*), 898(*s*), 822(*s*), 787(*s*), 732(*m*), 710(*m*), 687(*s*), 594(*m*), 539(*m*), 485(*m*), 453(w).

S2. Refinement

Hydrogen atoms bonded to C atoms of the 5-hydroxynicotinate anions were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). H atoms of the hydroxy functions were found from difference maps and were included in the refinement as riding atoms, with O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(O).

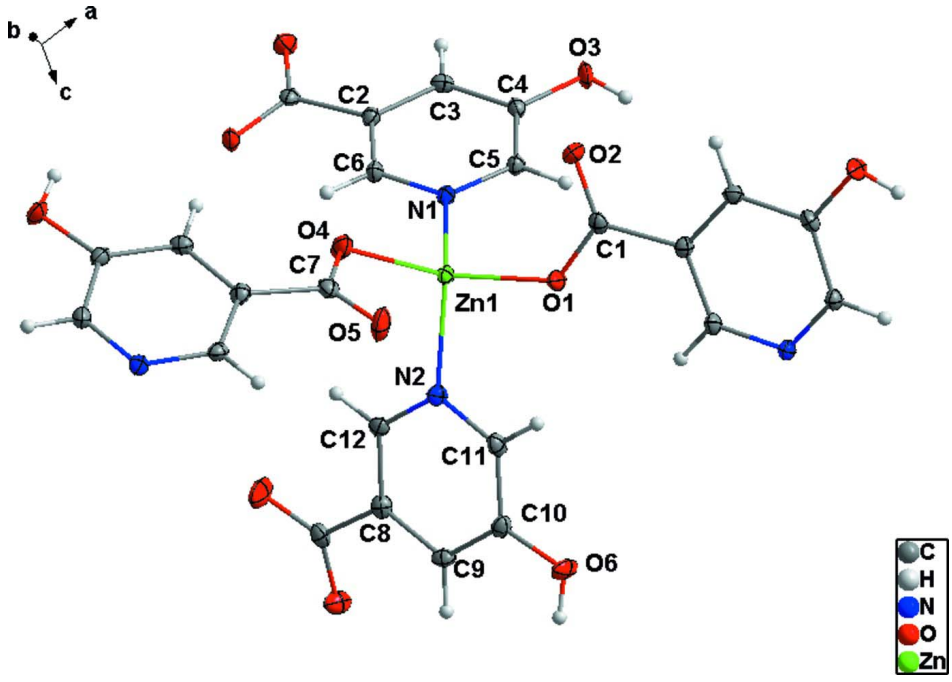


Figure 1

The coordination environment of the Zn^{II} ion in the title compound, showing displacement ellipsoids at the 50% probability level.

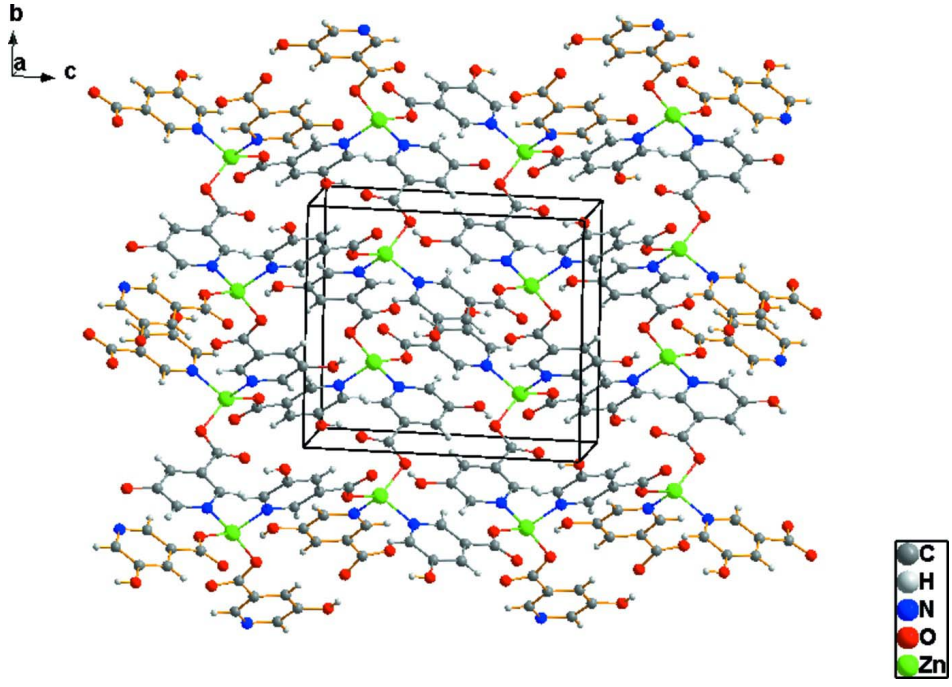


Figure 2

The packing in the structure of $[Zn(C_6H_4O_3N)_2]_n$, showing the polymeric character of the title compound.

Poly[bis(μ_2-5-hydroxynicotinato-κ^2N:O^3)zinc]

Crystal data

$[Zn(C_6H_4NO_3)_2]$
M_r = 341.57
Monoclinic, $P2_1/n$
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b = 10.5453 (7) Å
c = 12.6914 (8) Å
β = 104.640 (7)°
V = 1221.07 (14) Å^3
Z = 4
$F(000)$ = 688
D_x = 1.858 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
θ = 3.0–29.1°
μ = 2.04 mm^{-1}
T = 150 K
Block, yellow
0.41 × 0.37 × 0.17 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.488, T_{max} = 0.723
7054 measured reflections
2891 independent reflections
2397 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 29.3°, θ_{min} = 2.4°
h = −12→12
k = −14→12
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.075
S = 1.07
3345 reflections
192 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0271P)^2 + 0.5488P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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}
C1	0.8597 (2)	0.3757 (2)	0.30816 (18)	0.0111 (5)
C2	0.4890 (2)	0.1376 (2)	−0.09332 (18)	0.0104 (5)
C3	0.6200 (2)	0.0759 (2)	−0.08906 (19)	0.0118 (5)
H3	0.6325	0.0305	−0.1489	0.014*
C4	0.7328 (2)	0.0828 (2)	0.00604 (19)	0.0114 (5)

C5	0.7092 (2)	0.1514 (2)	0.09361 (19)	0.0114 (5)
H5	0.7828	0.1540	0.1581	0.014*
C6	0.4744 (2)	0.2077 (2)	−0.00397 (19)	0.0123 (5)
H6	0.3875	0.2512	−0.0078	0.015*
C7	0.3884 (2)	0.5320 (2)	0.19736 (19)	0.0135 (5)
C8	0.2504 (2)	0.1059 (2)	0.34892 (19)	0.0121 (5)
C9	0.3220 (3)	0.0977 (2)	0.45873 (19)	0.0122 (5)
H9	0.2824	0.0509	0.5065	0.015*
C10	0.4545 (3)	0.1615 (2)	0.49542 (19)	0.0126 (5)
C11	0.5125 (3)	0.2258 (2)	0.42054 (19)	0.0131 (5)
H11	0.6029	0.2656	0.4450	0.016*
C12	0.3118 (3)	0.1758 (2)	0.27914 (19)	0.0117 (5)
H12	0.2610	0.1836	0.2064	0.014*
N1	0.5831 (2)	0.21429 (19)	0.08815 (15)	0.0108 (4)
N2	0.4436 (2)	0.2330 (2)	0.31413 (15)	0.0122 (4)
O1	0.74133 (17)	0.32799 (17)	0.32074 (13)	0.0154 (4)
O2	0.87405 (18)	0.43130 (18)	0.22547 (13)	0.0179 (4)
O3	0.85858 (17)	0.02136 (18)	0.00882 (14)	0.0168 (4)
H3A	0.9134	0.0264	0.0702	0.025*
O4	0.41994 (18)	0.44819 (17)	0.13440 (13)	0.0178 (4)
O5	0.45953 (19)	0.55268 (19)	0.29152 (14)	0.0238 (5)
O6	0.53504 (19)	0.16272 (18)	0.60036 (13)	0.0188 (4)
H6A	0.4885	0.1288	0.6391	0.028*
Zn1	0.55375 (3)	0.32108 (3)	0.21439 (2)	0.01009 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0105 (10)	0.0118 (12)	0.0110 (11)	0.0024 (10)	0.0028 (9)	−0.0023 (9)
C2	0.0105 (10)	0.0102 (12)	0.0101 (11)	0.0001 (10)	0.0016 (9)	0.0015 (9)
C3	0.0130 (11)	0.0122 (13)	0.0113 (11)	−0.0013 (10)	0.0050 (9)	−0.0009 (10)
C4	0.0083 (10)	0.0120 (13)	0.0139 (12)	0.0006 (10)	0.0026 (9)	0.0016 (10)
C5	0.0097 (10)	0.0129 (13)	0.0104 (11)	0.0005 (10)	0.0006 (9)	0.0001 (9)
C6	0.0089 (11)	0.0160 (13)	0.0111 (12)	0.0011 (10)	0.0008 (9)	0.0005 (9)
C7	0.0107 (11)	0.0143 (13)	0.0158 (13)	−0.0011 (10)	0.0039 (10)	0.0019 (10)
C8	0.0122 (11)	0.0104 (12)	0.0132 (12)	0.0004 (10)	0.0021 (9)	−0.0021 (9)
C9	0.0154 (11)	0.0100 (12)	0.0122 (11)	−0.0003 (10)	0.0058 (9)	0.0001 (9)
C10	0.0129 (11)	0.0143 (13)	0.0097 (11)	0.0017 (10)	0.0012 (9)	−0.0017 (9)
C11	0.0100 (11)	0.0159 (13)	0.0128 (12)	−0.0013 (10)	0.0016 (10)	−0.0029 (10)
C12	0.0123 (11)	0.0128 (13)	0.0102 (11)	0.0026 (10)	0.0034 (9)	−0.0011 (9)
N1	0.0097 (9)	0.0127 (11)	0.0093 (9)	0.0001 (8)	0.0011 (8)	−0.0007 (8)
N2	0.0121 (9)	0.0131 (11)	0.0113 (10)	−0.0014 (9)	0.0028 (8)	−0.0009 (8)
O1	0.0096 (8)	0.0246 (10)	0.0109 (8)	−0.0041 (7)	0.0006 (7)	0.0016 (7)
O2	0.0157 (8)	0.0280 (11)	0.0097 (8)	0.0012 (8)	0.0025 (7)	0.0045 (7)
O3	0.0092 (8)	0.0250 (10)	0.0144 (9)	0.0079 (8)	−0.0002 (7)	−0.0027 (8)
O4	0.0190 (9)	0.0203 (10)	0.0136 (9)	0.0090 (8)	0.0029 (7)	0.0021 (7)
O5	0.0191 (9)	0.0302 (12)	0.0164 (10)	0.0070 (8)	−0.0060 (8)	−0.0029 (8)
O6	0.0184 (9)	0.0295 (12)	0.0071 (8)	−0.0073 (8)	0.0008 (7)	−0.0013 (7)

Zn1	0.00810 (14)	0.01335 (17)	0.00817 (15)	0.00045 (11)	0.00084 (10)	−0.00090 (11)

Geometric parameters (Å, º)

C1—O2	1.239 (3)	C8—C12	1.387 (3)
C1—O1	1.270 (3)	C8—C9	1.389 (3)
C1—C2^{i}	1.516 (3)	C8—C7^{iv}	1.509 (3)
C2—C3	1.385 (3)	C9—C10	1.392 (3)
C2—C6	1.390 (3)	C9—H9	0.9300
C2—C1^{ii}	1.516 (3)	C10—O6	1.356 (3)
C3—C4	1.395 (3)	C10—C11	1.387 (3)
C3—H3	0.9300	C11—N2	1.345 (3)
C4—O3	1.344 (3)	C11—H11	0.9300
C4—C5	1.390 (3)	C12—N2	1.351 (3)
C5—N1	1.348 (3)	C12—H12	0.9300
C5—H5	0.9300	N1—Zn1	2.034 (2)
C6—N1	1.348 (3)	N2—Zn1	2.052 (2)
C6—H6	0.9300	O1—Zn1	1.9364 (15)
C7—O5	1.233 (3)	O3—H3A	0.8200
C7—O4	1.276 (3)	O4—Zn1	1.9421 (17)
C7—C8^{iii}	1.509 (3)	O6—H6A	0.8200
O2—C1—O1	125.5 (2)	C10—C9—H9	120.9
O2—C1—C2^{i}	120.3 (2)	O6—C10—C11	116.6 (2)
O1—C1—C2^{i}	114.1 (2)	O6—C10—C9	124.5 (2)
C3—C2—C6	119.3 (2)	C11—C10—C9	118.9 (2)
C3—C2—C1^{ii}	120.8 (2)	N2—C11—C10	122.8 (2)
C6—C2—C1^{ii}	119.8 (2)	N2—C11—H11	118.6
C2—C3—C4	119.1 (2)	C10—C11—H11	118.6
C2—C3—H3	120.4	N2—C12—C8	121.7 (2)
C4—C3—H3	120.4	N2—C12—H12	119.2
O3—C4—C5	123.2 (2)	C8—C12—H12	119.2
O3—C4—C3	118.2 (2)	C5—N1—C6	119.1 (2)
C5—C4—C3	118.5 (2)	C5—N1—Zn1	121.74 (15)
N1—C5—C4	122.2 (2)	C6—N1—Zn1	119.12 (16)
N1—C5—H5	118.9	C11—N2—C12	118.4 (2)
C4—C5—H5	118.9	C11—N2—Zn1	116.97 (15)
N1—C6—C2	121.7 (2)	C12—N2—Zn1	124.49 (16)
N1—C6—H6	119.2	C1—O1—Zn1	127.16 (15)
C2—C6—H6	119.2	C4—O3—H3A	109.5
O5—C7—O4	125.0 (2)	C7—O4—Zn1	111.97 (15)
O5—C7—C8^{iii}	119.4 (2)	C10—O6—H6A	109.5
O4—C7—C8^{iii}	115.5 (2)	O1—Zn1—O4	134.17 (8)
C12—C8—C9	119.9 (2)	O1—Zn1—N1	106.71 (7)
C12—C8—C7^{iv}	119.2 (2)	O4—Zn1—N1	99.80 (8)
C9—C8—C7^{iv}	120.8 (2)	O1—Zn1—N2	95.90 (7)

C8—C9—C10	118.2 (2)	O4—Zn1—N2	105.76 (8)
C8—C9—H9	120.9	N1—Zn1—N2	115.24 (8)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O5^{v}	0.82	1.88	2.697 (2)	175
O6—H6*A*···O2^{vi}	0.82	1.83	2.651 (3)	174

Symmetry codes: (v) $-x+3/2, y-1/2, -z+1/2$; (vi) $x-1/2, -y+1/2, z+1/2$.