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Bis[4-(dimethylamino)pyridine-κN]-bis(salicylato-κO)zinc(II)

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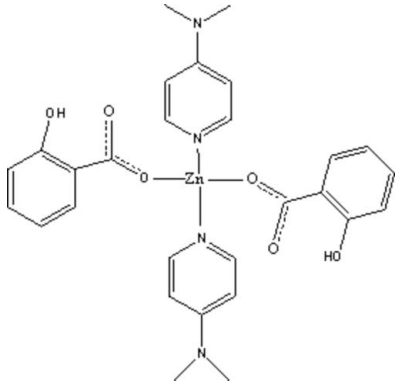
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.042; wR factor = 0.107; data-to-parameter ratio = 13.7.

The reaction of $Zn(CH_3COO)_2$ with 4-(dimethylamino)-pyridine (DMAP) and salicylic acid in a 1:2:2 molar ratio affords the title complex, $[Zn(C_7H_5O_3)_2(C_7H_{10}N_2)_2]$. The Zn^{II} atom resides on a twofold rotation axis and is tetrahedrally coordinated by two DMAP ligands and two salicylate anions in a distorted tetrahedral geometry.

Related literature

For related literature, see: Fu (2000); Tyrra *et al.* (2003); Wang *et al.* (2006).



Experimental

Crystal data

$[Zn(C_7H_5O_3)_2(C_7H_{10}N_2)_2]$
M_r = 583.93
Monoclinic, $C2/c$
a = 19.961 (2) Å
b = 7.5009 (9) Å
c = 18.488 (2) Å
β = 93.583 (8)°
V = 2762.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.94 mm^{-1}
T = 293 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEXII diffractometer
Absorption correction: none
6850 measured reflections
2419 independent reflections
2032 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.107
S = 1.04
2419 reflections
177 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2	0.82	1.79	2.522 (5)	148

Data collection: *APEX2* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2121).

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Fu, G.-C. (2000). *Acc. Chem. Res.* **33**, 412–420.
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Wang, H., Li, M.-X., Shao, M. & He, X. (2006). *Acta Cryst.* E**62**, m3309–m3311.

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supplementary materials

Acta Cryst. (2007). E**63**, m1930 [**doi:10.1107/S1600536807028322**]

Bis[4-(dimethylamino)pyridine-*κN*]bis(salicylato-*κO*)zinc(II)

K.-H. Lin, F.-F. Zhang, Z.-Y. Yu and S. Min

Comment

In preparing metal complexes, carboxylate and pyridine ligands have been frequently employed (Wang *et al.*, 2006). Although 4-dimethylaminopyridine (DMAP) has good coordination ability, there are few reports on its complexes (Tyrra *et al.*, 2003) except a lot of reports on its nucleophilic catalytic properties (Fu, 2000). We report here the synthesis and crystal structure of a new DMAP mononuclear zinc(II) complex.

The asymmetric unit contains one-half molecule of $[Zn(C_7H_{10}N_2)_2(C_7H_5O_3)_2]$ (Fig. 1). The Zn atom resides on a site of 2-fold symmetry and is in a tetrahedral environment with the Zn1—O1 and Zn1—N1 bond lengths 2.002 (3) and 2.028 (2) Å, respectively. The DMAP ligands coordinate to zinc through the pyridyl N atoms while the salicylate anions bond through a single carboxyl O atom. The non-coordinated carboxyl O atom is involved in an intramolecular hydrogen bond to the non-coordinated hydroxyl group (Table 1).

Experimental

An aqueous solution (10 ml) containing salicylic acid (0.1381 g, 1.0 mmol) and $Zn(CH_3COO)_2{\cdot}2H_2O$ (0.1098 g, 0.5 mmol) was mixed and refluxed for 1 h. Then another ethanol solution (5 ml) of DMAP (0.1222 g, 1 mmol) was added dropwise into the above solution. The resulted mixture was refluxed for 4 h. The solution was filtered after cooling to room temperature. Colorless single crystals suitable for X-ray diffraction were obtained from the filtrate after 4 d.

Refinement

The H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances = 0.93Å and $U_{iso}(H) = 1.2U_{eq}(C)$, O—H distance = 0.8156 Å and $U_{iso}(H) = 1.2U_{eq}(O)$

Figures

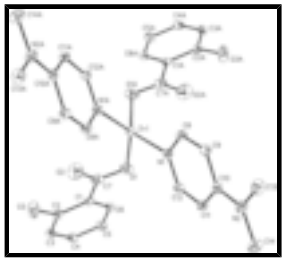


Fig. 1. The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

Bis[4-(dimethylamino)pyridine-κ*N*]bis(salicylato-κ*O*)zinc(II)

Crystal data

$[Zn(C_7H_5O_3)_2(C_7H_{10}N_2)_2]$	F_{000} = 1216
M_r = 583.93	D_x = 1.404 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
a = 19.961 (2) Å	Cell parameters from 2692 reflections
b = 7.5009 (9) Å	θ = 2.2–23.7º
c = 18.488 (2) Å	μ = 0.94 mm^{-1}
β = 93.583 (8)º	*T* = 293 (2) K
V = 2762.7 (5) Å^3	Block, colourless
Z = 4	0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEXII diffractometer	2032 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.020
Monochromator: graphite	θ_{max} = 25.0º
T = 293(2) K	θ_{min} = 2.2º
φ and ω scans	*h* = −23→20
Absorption correction: none	*k* = −8→8
6850 measured reflections	*l* = −21→21
2419 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.042	H-atom parameters constrained
$wR(F^2)$ = 0.107	$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 3.8555P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
2419 reflections	$\Delta\rho_{max}$ = 0.58 e Å^{-3}
177 parameters	$\Delta\rho_{min}$ = −0.29 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	0.0000	0.58617 (6)	0.2500	0.05591 (19)
C1	0.15627 (14)	0.2514 (4)	0.31399 (16)	0.0536 (7)
C2	0.19655 (18)	0.2440 (5)	0.37794 (17)	0.0682 (9)
C3	0.24291 (17)	0.1075 (5)	0.38911 (19)	0.0709 (9)
H3	0.2699	0.1028	0.4319	0.085*
C4	0.24888 (16)	−0.0184 (5)	0.3378 (2)	0.0734 (9)
H4	0.2803	−0.1091	0.3456	0.088*
C5	0.20952 (17)	−0.0147 (5)	0.2746 (2)	0.0783 (10)
H5	0.2138	−0.1026	0.2397	0.094*
C6	0.16372 (15)	0.1199 (4)	0.26335 (18)	0.0642 (8)
H6	0.1370	0.1223	0.2203	0.077*
C7	0.10649 (19)	0.3990 (5)	0.3030 (3)	0.0789 (10)
C8	−0.01148 (16)	0.9075 (4)	0.15743 (17)	0.0649 (8)
H8	−0.0528	0.9157	0.1778	0.078*
C9	0.00609 (16)	1.0410 (4)	0.11356 (16)	0.0625 (8)
H9	−0.0228	1.1372	0.1051	0.075*
C10	0.06755 (15)	1.0362 (4)	0.08056 (14)	0.0532 (7)
C11	0.10614 (15)	0.8820 (4)	0.09559 (16)	0.0566 (7)
H11	0.1466	0.8665	0.0740	0.068*
C12	0.08467 (14)	0.7553 (4)	0.14149 (15)	0.0543 (7)
H12	0.1122	0.6570	0.1510	0.065*
C13	0.0484 (2)	1.3291 (5)	0.0261 (2)	0.0835 (11)
H13A	0.0519	1.4051	0.0670	0.100*
H13B	0.0638	1.3916	−0.0158	0.100*
H13C	0.0013	1.2988	0.0151	0.100*
C14	0.1501 (2)	1.1575 (6)	0.0028 (2)	0.0967 (13)
H14A	0.1476	1.0874	−0.0415	0.116*
H14B	0.1679	1.2744	−0.0092	0.116*
H14C	0.1871	1.1006	0.0343	0.116*
N1	0.02661 (12)	0.7628 (3)	0.17384 (12)	0.0555 (6)
N2	0.08845 (14)	1.1690 (4)	0.03907 (14)	0.0683 (7)
O1	0.07388 (13)	0.4073 (3)	0.24406 (18)	0.0930 (8)
O2	0.09803 (19)	0.5057 (5)	0.3525 (2)	0.1405 (14)
O3	0.1925 (2)	0.3681 (5)	0.43048 (16)	0.1324 (13)
H3A	0.1616	0.4356	0.4193	0.159*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0570 (3)	0.0546 (3)	0.0565 (3)	0.000	0.0065 (2)	0.000
C1	0.0468 (15)	0.0503 (17)	0.0651 (18)	−0.0040 (13)	0.0138 (13)	0.0031 (14)
C2	0.079 (2)	0.069 (2)	0.0574 (19)	−0.0070 (8)	0.0153 (16)	−0.0098 (16)
C3	0.065 (2)	0.083 (2)	0.063 (2)	−0.0058 (18)	−0.0032 (15)	0.0084 (18)
C4	0.0552 (19)	0.071 (2)	0.094 (3)	0.0100 (17)	0.0049 (18)	0.003 (2)
C5	0.070 (2)	0.075 (2)	0.089 (3)	0.0123 (19)	0.0035 (19)	−0.027 (2)
C6	0.0551 (18)	0.075 (2)	0.0624 (18)	−0.0047 (16)	0.0005 (14)	−0.0051 (16)
C7	0.064 (2)	0.065 (2)	0.109 (3)	0.0015 (18)	0.017 (2)	0.016 (2)
C8	0.0542 (17)	0.075 (2)	0.0675 (19)	0.0148 (16)	0.0160 (14)	0.0046 (17)
C9	0.0636 (19)	0.0623 (19)	0.0620 (19)	0.0193 (15)	0.0068 (15)	0.0026 (15)
C10	0.0603 (17)	0.0541 (17)	0.0449 (15)	0.0057 (13)	0.0027 (13)	−0.0044 (13)
C11	0.0536 (16)	0.0557 (18)	0.0617 (17)	0.0072 (13)	0.0126 (13)	0.0016 (14)
C12	0.0521 (16)	0.0525 (17)	0.0583 (17)	0.0098 (13)	0.0047 (13)	−0.0018 (13)
C13	0.108 (3)	0.061 (2)	0.083 (2)	0.018 (2)	0.011 (2)	0.0116 (19)
C14	0.097 (3)	0.086 (3)	0.111 (3)	0.005 (2)	0.039 (2)	0.029 (2)
N1	0.0513 (13)	0.0581 (15)	0.0575 (14)	0.0068 (11)	0.0074 (11)	0.0015 (11)
N2	0.0818 (18)	0.0587 (16)	0.0663 (16)	0.0107 (14)	0.0193 (14)	0.0111 (13)
O1	0.0681 (15)	0.0830 (18)	0.127 (2)	0.0055 (13)	−0.0008 (16)	0.0269 (16)
O2	0.159 (3)	0.104 (2)	0.081 (3)	0.043 (3)	0.006 (3)	−0.030 (2)
O3	0.187 (4)	0.121 (3)	0.087 (2)	0.036 (2)	−0.008 (2)	−0.0484 (19)

Geometric parameters (Å, °)

Zn1—O1^{i}	2.002 (3)	C8—C9	1.349 (4)
Zn1—O1	2.002 (3)	C8—H8	0.9300
Zn1—N1	2.028 (2)	C9—C10	1.404 (4)
Zn1—N1^{i}	2.028 (2)	C9—H9	0.9300
C1—C6	1.375 (4)	C10—N2	1.340 (4)
C1—C2	1.389 (4)	C10—C11	1.408 (4)
C1—C7	1.493 (5)	C11—C12	1.361 (4)
C2—O3	1.351 (4)	C11—H11	0.9300
C2—C3	1.387 (5)	C12—N1	1.338 (3)
C3—C4	1.349 (5)	C12—H12	0.9300
C3—H3	0.9300	C13—N2	1.454 (4)
C4—C5	1.367 (5)	C13—H13A	0.9460
C4—H4	0.9300	C13—H13B	0.9719
C5—C6	1.369 (5)	C13—H13C	0.9775
C5—H5	0.9300	C14—N2	1.440 (4)
C6—H6	0.9300	C14—H14A	0.9714
C7—O1	1.235 (5)	C14—H14B	0.9769
C7—O2	1.236 (5)	C14—H14C	1.0073
C8—N1	1.349 (4)	O3—H3A	0.8156
O1^{i}—Zn1—O1	95.82 (15)	C8—C9—H9	119.7
O1^{i}—Zn1—N1	134.57 (12)	C10—C9—H9	119.7

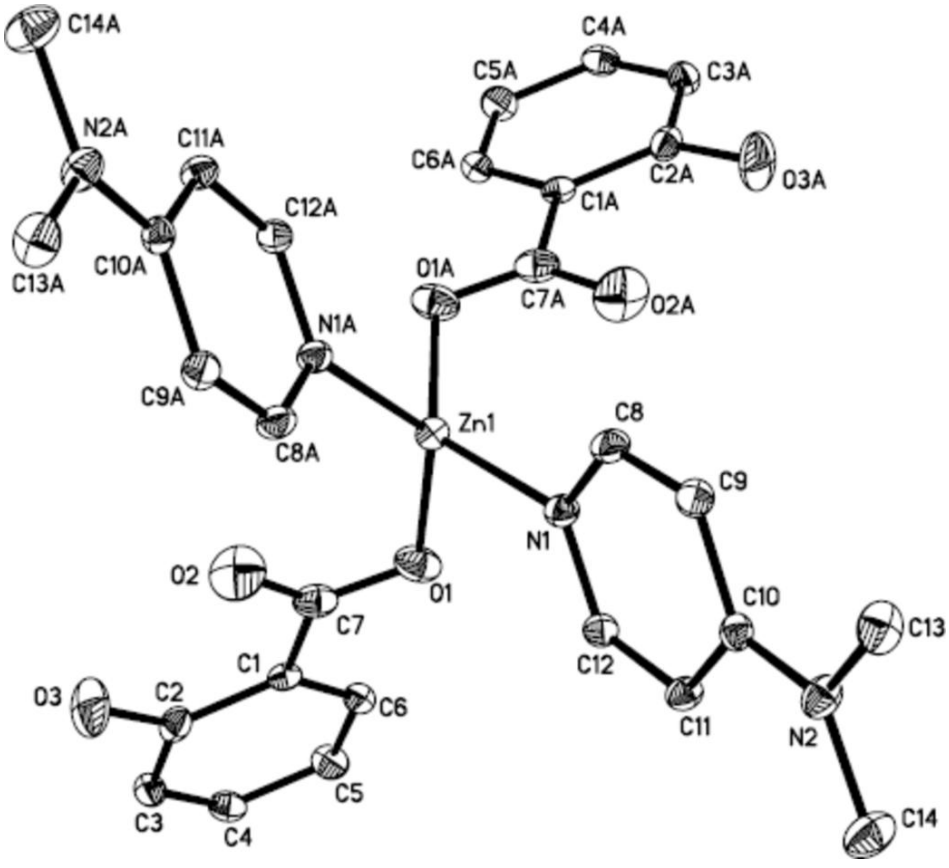
O1—Zn1—N1	100.02 (10)	N2—C10—C9	122.8 (3)
O1^{i}—Zn1—N1^{i}	100.02 (10)	N2—C10—C11	122.5 (3)
O1—Zn1—N1^{i}	134.57 (12)	C9—C10—C11	114.7 (3)
N1—Zn1—N1^{i}	98.43 (14)	C12—C11—C10	120.5 (3)
C6—C1—C2	118.0 (3)	C12—C11—H11	119.8
C6—C1—C7	122.6 (3)	C10—C11—H11	119.8
C2—C1—C7	119.4 (3)	N1—C12—C11	124.4 (3)
O3—C2—C3	118.1 (3)	N1—C12—H12	117.8
O3—C2—C1	121.8 (3)	C11—C12—H12	117.8
C3—C2—C1	120.1 (3)	N2—C13—H13A	110.7
C4—C3—C2	120.0 (3)	N2—C13—H13B	109.4
C4—C3—H3	120.0	H13A—C13—H13B	109.7
C2—C3—H3	120.0	N2—C13—H13C	110.7
C3—C4—C5	121.1 (3)	H13A—C13—H13C	109.2
C3—C4—H4	119.5	H13B—C13—H13C	107.1
C5—C4—H4	119.5	N2—C14—H14A	115.1
C4—C5—C6	119.1 (3)	N2—C14—H14B	112.7
C4—C5—H5	120.4	H14A—C14—H14B	107.2
C6—C5—H5	120.4	N2—C14—H14C	111.9
C5—C6—C1	121.7 (3)	H14A—C14—H14C	104.8
C5—C6—H6	119.1	H14B—C14—H14C	104.4
C1—C6—H6	119.1	C12—N1—C8	115.2 (3)
O1—C7—O2	122.1 (4)	C12—N1—Zn1	123.7 (2)
O1—C7—C1	117.9 (4)	C8—N1—Zn1	120.88 (19)
O2—C7—C1	120.0 (4)	C10—N2—C14	121.9 (3)
N1—C8—C9	124.6 (3)	C10—N2—C13	121.4 (3)
N1—C8—H8	117.7	C14—N2—C13	116.7 (3)
C9—C8—H8	117.7	C7—O1—Zn1	109.6 (3)
C8—C9—C10	120.6 (3)	C2—O3—H3A	108.9

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···O2	0.82	1.79	2.522 (5)	148

Fig. 1


C14A
N2A
C11A
C13A
C10A
C12A
N1A
C9A
C8A
C4A
C5A
C3A
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C2A
O3A
C1A
O1A
C7A
O2A
Zn1
C8
C9
N1
O2
O1
C7
C10
C13
C12
C1
O3
C2
C6
C11
N2
C3
C5
C4
C14