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Pyridine{4,4′,6,6′-tetrabromo-2,2′-[1,2-phenylenebis(nitrilomethylidyne)]diphenolato}zinc(II) dimethylformamide solvate

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.009 Å
R factor = 0.045
wR factor = 0.162
Data-to-parameter ratio = 16.0

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

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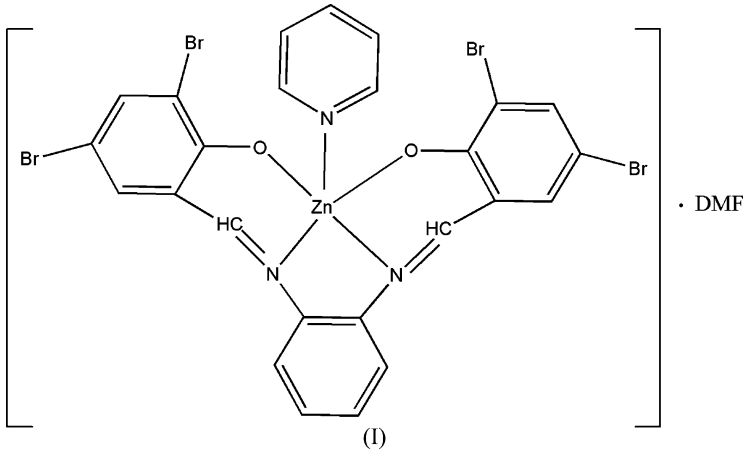
The tetradentate Schiff base ligand derived from the condensation of 3,5-dibromosalicylaldehyde and 1,2-phenylenediamine, in the presence of pyridine, forms a square-pyramidal five-coordinate Zn complex, $[Zn(C_{20}H_{10}Br_4N_2O_2)(C_5H_5N)]\cdot C_3H_7N0$.

Comment

The crystal structure and some properties of the 1,2-*N*,*N*-disalicylidenephenylaminato–nickel(II) complex were previously reported by Bei *et al.* (2003). We report here the synthesis and crystal structure of a novel complex, namely pyridine{4,4′,6,6′-tetrabromo-2,2′-[1,2-phenylenebis(nitrilomethylidyne)]diphenolato}zinc(II) dimethylformamide solvate, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The Zn atom has a distorted square-pyramidal coordination geometry, namely two N atoms and two O atoms from one Schiff base ligand and one N atom of the pyridine molecule. The Zn atom is displaced by 0.3633 (6) Å out of the basal plane defined by atoms N1, N2, O2 and O1. The crystal structure is stabilized by hydrogen bonds of the type C—H···O (Table 2 and Fig. 2). C21—H21···O1 is an internal contact which is omitted from Fig. 2.

Experimental

The title complex was synthesized in two stages. In the first stage, 3,5-dibromosalicylaldehyde was prepared according to Elzbieta *et al.* (1964). To ethanol (100 ml) containing 1,2-phenylenediamine (6 g), two mole equivalents of 3,5-dibromosalicylaldehyde in ethanol (50 ml) were slowly added with continuous stirring; the Schiff base molecule, *viz*. 4,4′,6,6′-tetrabromo-2,2′-[1,2-phenylenebis(nitrilomethylidyne)]diphenol, precipitated immediately. In the second

stage, the ligand (1 mmol), $Zn(OAc)_2$ (1 mmol), DMF (30 ml) and pyridine (10 ml) were refluxed for 1 h. The hot solution was filtered and allowed to stand at room temperature undisturbed for about three weeks, resulting in yellow crystals.

Crystal data

$[Zn(C_{20}H_{10}Br_4N_2O_2)(C_5H_5N)]\cdot C_3H_7N0$
$M_r = 847.51$
Monoclinic, $P2_1/c$
$a = 8.2289$ (9) Å
$b = 15.2809$ (17) Å
$c = 23.793$ (3) Å
$\beta = 95.946$ (2)°
$V = 2975.7$ (6) Å^3
$Z = 4$
$D_x = 1.892$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3507 reflections
$\theta = 2.5$–22.7°
$\mu = 6.23$ mm^{-1}
$T = 292$ (2) K
Block, yellow
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.300$, $T_{max} = 0.326$
15973 measured reflections
5823 independent reflections
3982 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 26.0°$
$h = -10 \rightarrow 9$
$k = -18 \rightarrow 18$
$l = -26 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.162$
$S = 1.04$
5823 reflections
363 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0926P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.83$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O2	1.980 (4)	Zn1—N1	2.104 (4)
Zn1—O1	1.993 (4)	Zn1—N2	2.123 (5)
Zn1—N3	2.082 (4)		
O2—Zn1—O1	95.34 (16)	N3—Zn1—N1	109.37 (18)
O2—Zn1—N3	105.65 (18)	O2—Zn1—N2	88.31 (17)
O1—Zn1—N3	95.87 (18)	O1—Zn1—N2	160.50 (18)
O2—Zn1—N1	144.13 (17)	N3—Zn1—N2	101.58 (18)
O1—Zn1—N1	88.70 (17)	N1—Zn1—N2	77.42 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
C21—H21···O1	0.93	2.53	3.092 (7)	119
C24—H24···O2^{i}	0.93	2.56	3.275 (8)	134
C12—H12···O3^{ii}	0.93	2.48	3.395 (11)	168

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

The H atoms were placed in calculated positions and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$ and C—H distances of 0.93 Å.

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

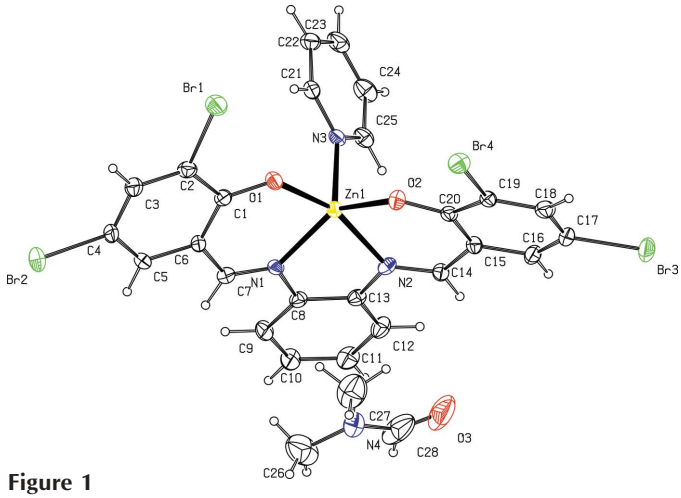


Figure 1
View of the title complex, showing labelling of the non-H atoms and 20% probability ellipsoids.

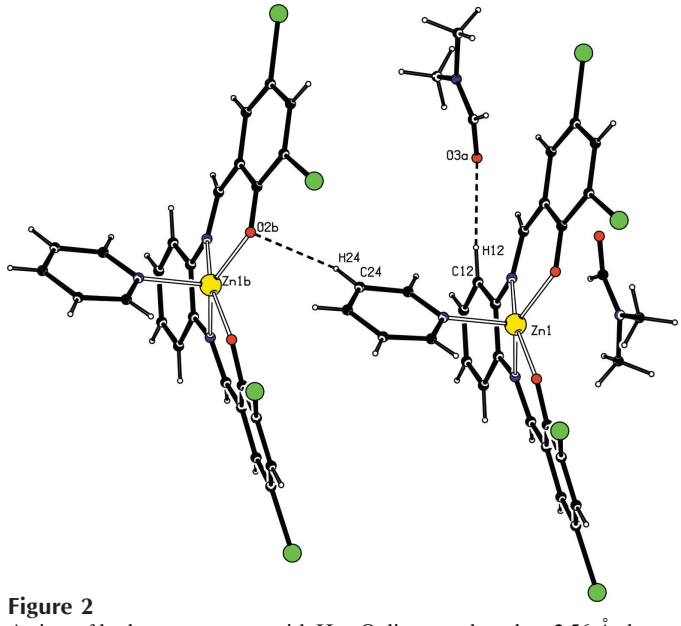


Figure 2
A view of hydrogen contacts with H···O distances less than 2.56 Å shown by dashed lines.

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