

Dichloro{2,4-dichloro-6-[2-(dimethylamino)-ethyliminomethyl]phenolato}zinc(II)

Zhong-Lu You

Department of Chemistry and Chemical
Engineering, Liaoning Normal University,
Dalian 116029, People's Republic of ChinaCorrespondence e-mail:
youzhonglu@yahoo.com.cn

Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.051
 wR factor = 0.098
Data-to-parameter ratio = 20.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title complex, $[\text{Zn}(\text{C}_{11}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O})\text{Cl}_2]$, is a mononuclear zinc(II) compound. The Zn^{II} atom is four-coordinated in a tetrahedral configuration by one imine N and one phenolate O atoms of a Schiff base ligand, and by two Cl atoms. The molecules are linked through intermolecular $\text{N}-\text{H}\cdots\text{Cl}$, $\text{C}-\text{H}\cdots\text{O}$, and $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds, forming a three-dimensional framework.

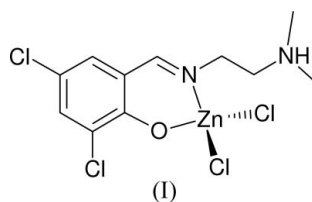
Received 12 October 2005

Accepted 25 October 2005

Online 31 October 2005

Comment

Transition metal complexes containing Schiff base ligands have been of great interest for many years (Yamada, 1999). These complexes play an important role in coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures (Lacroix, 1996; Goswami & Eichhorn, 1999; Rybak-Akimova *et al.*, 1998). The study of the variety of products in self-assembly processes between labile metal ions and flexible multidentate ligands is an interesting topic in supramolecular chemistry. Recently, the author has reported a mononuclear Schiff base zinc(II) compound, dichloro{2-[2-(dimethylamino)ethyliminomethyl]-4-nitrophenolato}zinc(II) (You, 2005). As an extension of the work on these complexes, the crystal structure of a similar complex, (I), is reported here.



Complex (I) is a mononuclear zinc(II) compound (Fig. 1). The Zn^{II} atom is four-coordinated in a tetrahedral configuration by one imine N and one phenolate O atoms of a Schiff base ligand, and by two Cl atoms. The ZnONCl_2 coordination forms a slightly distorted tetrahedral geometry, as usually observed in the structures of Zn^{II} complexes (McCleverty *et al.*, 1980; You *et al.*, 2003), with angles subtended at the Zn^{II} atom in the range $95.13(14)$ – $123.71(10)^\circ$ (Table 1). The $\text{Zn}-\text{O}$ and $\text{Zn}-\text{N}$ bond lengths are comparable with the corresponding values observed in other Schiff base zinc(II) complexes (Usman *et al.*, 2003; Erxleben, 2001; Chisholm *et al.*, 2001). The $\text{C}7=\text{N}1$ bond length (Table 1) conforms to the normal value for a $\text{C}=\text{N}$ double bond [$1.279(8)$ Å; Allen *et al.*, 1987].

In the crystal structure, the molecules are linked *via* intermolecular $\text{N}-\text{H}\cdots\text{Cl}$, $\text{C}-\text{H}\cdots\text{O}$, and $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen

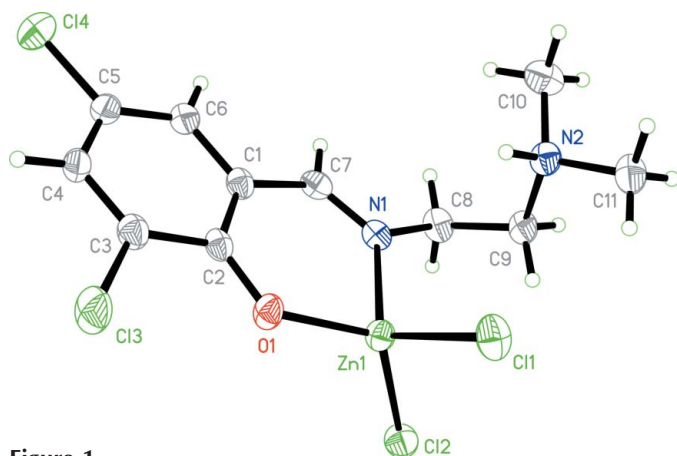


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

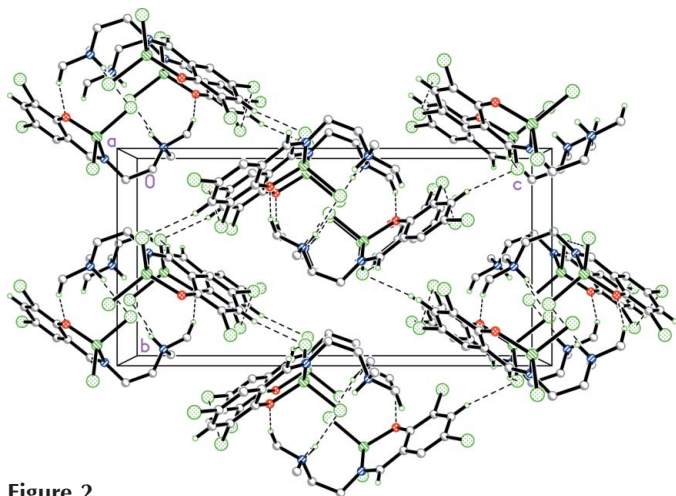


Figure 2
The crystal packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

bonds (Table 2), forming a three-dimensional framework (Fig. 2).

Experimental

3,5-Dichlorosalicylaldehyde (0.1 mmol, 19.1 mg) and *N,N'*-dimethylethane-1,2-diamine (0.1 mmol, 8.8 mg) were dissolved in MeOH (10 ml). The mixture was stirred at room temperature for 10 min to give a clear yellow solution. To this solution was added an MeOH solution (5 ml) of ZnCl₂ (0.1 mmol, 13.4 mg) with stirring. The resulting mixture was stirred for another 10 min at room temperature. After keeping the filtrate in air for 5 d, colourless needle-shaped crystals were formed at the bottom of the vessel.

Crystal data

[Zn(C₁₁H₁₄Cl₂N₂O)Cl₂]
M_r = 397.41
 Orthorhombic, *P*2₁2₁2₁
a = 7.728 (1) Å
b = 10.127 (1) Å
c = 20.237 (2) Å
V = 1583.8 (3) Å³
Z = 4
D_x = 1.667 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 3029 reflections
 θ = 2.2–23.0°
 μ = 2.22 mm⁻¹
T = 298 (2) K
 Fragment cut from needle, colourless
 0.17 × 0.14 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 T_{\min} = 0.704, T_{\max} = 0.879
 13624 measured reflections

3622 independent reflections
 3137 reflections with $I > 2\sigma(I)$
 R_{int} = 0.049
 θ_{max} = 27.5°
 h = -9 → 10
 k = -12 → 13
 l = -25 → 26

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.051
 $wR(F^2)$ = 0.098
 S = 1.14
 3622 reflections
 177 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}}$ = 0.001
 $\Delta\rho_{\text{max}}$ = 0.54 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -0.42 e Å⁻³
 Absolute structure: Flack (1983), 1526 Friedel pairs
 Flack parameter: 0.025 (17)

Table 1

Selected geometric parameters (Å, °).

Zn1—O1	1.926 (3)	Zn1—Cl1	2.2487 (14)
Zn1—N1	2.019 (4)	N1—C7	1.283 (6)
Zn1—Cl2	2.2427 (13)	N1—C8	1.458 (5)
O1—Zn1—N1	95.13 (14)	O1—Zn1—Cl1	110.26 (10)
O1—Zn1—Cl2	123.71 (10)	N1—Zn1—Cl1	115.15 (11)
N1—Zn1—Cl2	105.07 (11)	Cl2—Zn1—Cl1	107.31 (5)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N2—H2...Cl1 ⁱ	0.90 (4)	2.33 (2)	3.186 (4)	158 (5)
C4—H4...Cl2 ⁱⁱ	0.93	2.75	3.637 (4)	160
C7—H7...Cl2 ⁱⁱⁱ	0.93	2.80	3.655 (4)	154
C11—H11B...O1 ⁱ	0.96	2.40	3.307 (4)	157

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

Atom H2 was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å and with the $U_{\text{iso}}(\text{H})$ value fixed at 0.08 Å². All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å and $U_{\text{iso}}(\text{H})$ = 1.2 or 1.5 $U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; 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*.

The author thanks the Liaoning Normal University, People's Republic of China, for funding this study.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Bruker (1998). *SMART* (Version 5.628) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.

- Chisholm, M. H., Gallucci, J. C. & Zhen, H. (2001). *Inorg. Chem.* **40**, 5051–5054.
- Erxleben, A. (2001). *Inorg. Chem.* **40**, 208–213.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Goswami, N. & Eichhorn, D. M. (1999). *Inorg. Chem.* **38**, 4329–4333.
- Lacroix, P. G. (1996). *Chem. Mater.* **8**, 541–545.
- McCleverty, J. A., Morrison, N. J., Spencer, N., Ashworth, C. C., Bailey, N. A., Johnson, M. R., Smith, J. M. A., Tabbiner, B. A. & Taylor, C. R. (1980). *J. Chem. Soc. Dalton Trans.* pp. 1945–1957.
- Rybak-Akimova, E. V., Alcock, N. W. & Busch, D. H. (1998). *Inorg. Chem.* **37**, 1563–1574.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997a). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
- Usman, A., Fun, H.-K., Chantrapromma, S., Zhu, H.-L. & Liu, Q.-X. (2003). *Acta Cryst.* **E59**, o215–o217.
- Yamada, S. (1999). *Coord. Chem. Rev.* **190–192**, 537–555.
- You, Z.-L. (2005). *Acta Cryst.* **C61**, m383–m385.
- You, Z.-L., Lin, Y.-S., Liu, W.-S., Tan, M.-Y. & Zhu, H.-L. (2003). *Acta Cryst.* **E59**, m1025–m1027.