Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Dichloro(4′-phenyl-2,2′:6′,2″-terpyridine-κ^3N)-zinc(II) monohydrate

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Received 2 August 2004
Accepted 6 September 2004
Online 11 September 2004

Key indicators

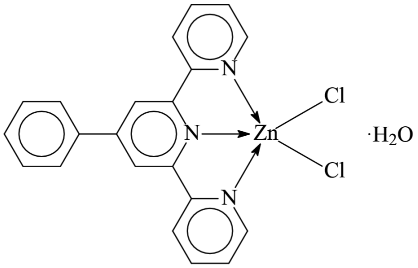
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
R factor = 0.062
wR factor = 0.181
Data-to-parameter ratio = 17.2

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

The Zn atom in each of the two symmetry-independent complex molecules of the title compound, $[ZnCl_2(C_{21}H_{15}N_3)]\cdot H_2O$, shows trigonal-bipyramidal coordination as the *N*-heterocycle is able to span the two axial sites of the coordination polyhedron. One of the molecules occupies a position in the crystal structure such that it does not interact with any water molecule; the other interacts with two water molecules to give rise to a hydrogen-bonded chain that runs along the *a* axis of the triclinic unit cell.

Comment

4′-Phenyl-2,2′:6′,2″-terpyridine is a tridentate chelating agent that binds to a number of metal atoms, as does the unsubstituted terpyridine heterocycle (Constable *et al.*, 1990). Terpyridine has been reported to form a five-coordinate complex with zinc dichloride (Vlasse *et al.*, 1983); the presence of the phenyl substituent leads to no significant differences in the geometry around the zinc atom (Fig. 1).



(I)

The asymmetric unit of the title compound, (I), consists of two crystallographically independent complexes and two water molecules; however, the two independent complexes exist in slightly different surroundings. One of the complexes has no interactions with water molecules, while the other forms water–chlorine (O—H···Cl) hydrogen bonds, giving rise to a chain motif running along the *a* axis. The coordination geometry of the Zn atom in both complexes is trigonal bipyramidal.

Experimental

To an aqueous solution (10 ml) of zinc dichloride (0.068 g, 0.5 mmol) was added 4′-phenyl-2,2′:6′,2″-terpyridine (0.15 g, 0.5 mmol), synthesized according to a procedure described by Constable *et al.* (1990). The mixture was placed in a 25 ml Teflon-lined Parr bomb and heated at 433 K for 38 h. The bomb was then cooled to room temperature at 5 K h^{-1}. Crystals were obtained in about 30% yield. Analysis calculated for $C_{21}H_{17}Cl_2N_3OZn$: C 54.36, H 3.66, N 9.06%; found: C 54.40, H 3.73, N 8.97%. IR (KBr, cm^{-1}): 3478 (*m*), 3065 (*w*), 1608 (*vs*), 1565 (*m*), 1556 (*m*), 1482 (*s*), 1417 (*s*), 1017 (*m*), 778 (*s*), 739 (*m*), 700 (*m*), 639 (*m*), 504 (*w*).

Crystal data

$[ZnCl_2(C_{21}H_{15}N_3)]\cdot H_2O$
$M_r = 463.65$
Triclinic, $P\overline{1}$
$a = 7.8813$ (6) Å
$b = 16.355$ (1) Å
$c = 17.228$ (1) Å
$\alpha = 117.131$ (1)°
$\beta = 95.273$ (1)°
$\gamma = 93.091$ (1)°
$V = 1956.4$ (2) Å^3
$Z = 4$
$D_x = 1.574$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2259 reflections
$\theta = 2.4$–19.4°
$\mu = 1.55$ mm^{-1}
$T = 298$ (2) K
Prism, orange
0.41 × 0.34 × 0.21 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.580$, $T_{\max} = 0.722$
16 985 measured reflections
8709 independent reflections
5414 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -20 \rightarrow 21$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.181$
$S = 1.03$
8709 reflections
505 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0885P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.78$ e Å^{-3}
$\Delta\rho_{\min} = -0.51$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—N1	2.180 (4)	Zn2—N4	2.191 (4)
Zn1—N2	2.104 (4)	Zn2—N5	2.095 (4)
Zn1—N3	2.189 (4)	Zn2—N6	2.159 (4)
Zn1—Cl1	2.271 (1)	Zn2—Cl3	2.274 (2)
Zn1—Cl2	2.256 (1)	Zn2—Cl4	2.273 (2)
N1—Zn1—N2	74.3 (2)	N4—Zn2—N5	74.1 (2)
N1—Zn1—N3	145.0 (2)	N4—Zn2—N6	148.1 (2)
N1—Zn1—Cl1	101.5 (1)	N4—Zn2—Cl3	98.0 (1)
N1—Zn1—Cl2	98.9 (1)	N4—Zn2—Cl4	98.8 (1)
N2—Zn1—N3	73.7 (2)	N5—Zn2—N6	74.8 (2)
N2—Zn1—Cl1	105.8 (1)	N5—Zn2—Cl3	114.9 (1)
N2—Zn1—Cl2	140.7 (1)	N5—Zn2—Cl4	134.9 (1)
N3—Zn1—Cl1	100.4 (1)	N6—Zn2—Cl3	101.2 (1)
N3—Zn1—Cl2	97.0 (1)	N6—Zn2—Cl4	98.2 (1)
Cl1—Zn1—Cl2	113.5 (1)	Cl3—Zn2—Cl4	110.2 (1)

H atoms attached to C atoms were placed in calculated positions [C—H 0.93 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C)], and were included in the refinement in riding-model approximation. One of the water molecules, O2*W*, shows large displacement parameters, indicating disorder. The H atoms of the water molecules were not located; they were placed in chemically sensible positions by using *HYDROGEN* (Nardelli, 1999) and their $U_{\rm iso}$ values were set at 1.2$U_{\rm eq}$(parent atom).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

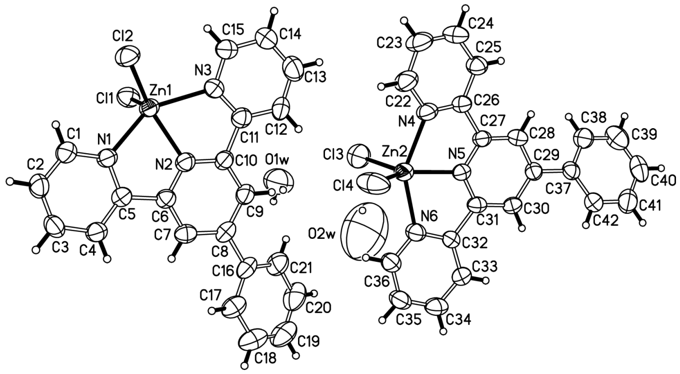


Figure 1
ORTEP (Johnson, 1976) plot of the two independent $[Zn(C_{21}H_{15}N_3Cl_2)]$ complexes and two water molecules, showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

The authors thank the Research Startup Foundation of Shantou University (No. 130 171045) and the University of Malaya for supporting this study.

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