Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

A second polymorph of dichloridobis(1,10-phenanthroline-κ^2N,N')tungsten(II)

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Received 16 March 2007
Accepted 20 March 2007

The title compound, $[WCl_2(C_{12}H_8N_2)_2]$, is a six-coordinate complex with a distorted octahedral geometry around the W atom. C—H···Cl hydrogen-bond interactions and π–π stacking interactions are responsible for the supramolecular assembly.

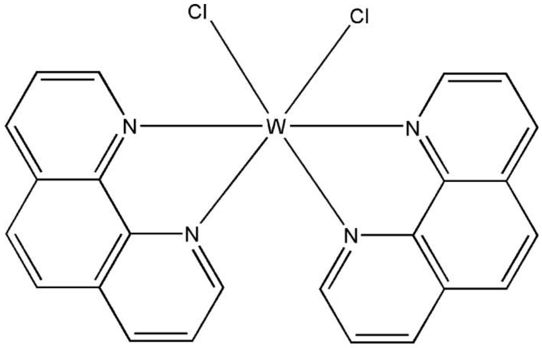
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.009 Å
R factor = 0.029
wR factor = 0.067
Data-to-parameter ratio = 17.4

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

Comment

We recently reported the structure of the tungsten complex dichloridobis(1,10-phenanthroline-κ^2N,N')tungsten(II), (II), which crystallizes in space group $P2_1/c$ (Xu & Lin, 2007). On repeating the experiment recently, to our surprise, we found a new polymorph, (I), that had crystallized in the space group $P\overline{1}$.



(I)

The structure of (I) is very similar to the previously reported complex. The W atom is coordinated by four N atoms of two phenanthroline and two Cl ligands, forming a distorted octahedron (Fig. 1 and Table 1). The W—N2 bond [2.701 (4) Å] is longer than the three other W—N bonds [2.430 (4)–2.448 (4) Å] and those of the W–phen complexes reported previously (Lang *et al.*, 1994; Xu & Lin, 2007). The Cl atoms are *cis* to one another, with Cl1—W—Cl2 = 107.6 (1)°, and the W—Cl distances show no significant differences from those of other tungsten(II) complexes (Junk & Atwood, 1997; Jaeger *et al.*, 2000). The bond lengths and angles within both phenathroline ligands are normal. The phenanthroline ligands are each nearly planar and make a dihedral angle of 67.69 (7)°. The phen ligand containing atoms N1 and N2 makes π–π stacking interactions with two symmetry-related phen neighbors (symmetry code: $-x, -y+2, -z+2$) (interplanar distance 3.38 Å), whereas the phen ligand containing atoms N3 and N4 interacts with a symmetry-related phen ligand [at $(-x+1, -y+1, -z+1)$; interplanar distance 3.38 Å]. Such π–π stacking interactions are responsible for the supramolecular assembly of the complex molecules into layers parallel to (100) (Fig. 2). These layers are further reinforced

doi:10.1107/S1600536807013414

and assembled by intermolecular C—H···Cl hydrogen bonds, generating a three-dimensional framework along the [100] direction.

Experimental

An evacuated silica tube (Φ_{outer} = 15 mm, D = 1 mm, L = 90 mm) charged with 0.326 g (1 mmol) WCl_4 (Aldrich >98%) was heated in a 915/925 K temperature gradient for one week. A small amount of black powder was formed in the low-temperature zone and a few black plate-shaped crystals (WCl_5) in the high-temperature zone. The black powder was removed from the ampoule into 30 ml DMF, and 0.180 g (1 mmol) phenanthroline was added to the solution. It was stirred for 30 min, then filtered to give a brown solution, which was maintained at 323 K. Colorless crystals grew after two weeks.

Crystal data

$[WCl_2(C_{12}H_8N_2)_2]$
M_r = 615.16
Triclinic, $P\overline{1}$
a = 9.3937 (19) Å
b = 9.7464 (19) Å
c = 12.739 (3) Å
α = 82.64 (3)°
β = 76.39 (3)°
γ = 71.84 (3)°
V = 1075.2 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 5.64 mm^{-1}
T = 295 (2) K
0.28 × 0.22 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995) T_{min} = 0.236, T_{max} = 0.326
10617 measured reflections
4865 independent reflections
4294 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.067
S = 1.05
4865 reflections
280 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.08 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

W1—N1	2.430 (4)	W1—N4	2.435 (4)
W1—N2	2.701 (4)	W1—Cl1	2.5829 (16)
W1—N3	2.448 (3)	W1—Cl2	2.4460 (15)
N1—W1—N3	85.67 (13)	N4—W1—N3	67.99 (12)
N1—W1—N4	138.03 (13)	N4—W1—Cl1	115.22 (10)
N1—W1—Cl1	93.09 (10)	N4—W1—Cl2	94.47 (9)
N1—W1—Cl2	106.23 (10)	Cl2—W1—N3	161.95 (10)
N3—W1—Cl1	84.79 (9)	Cl2—W1—Cl1	107.56 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3A···Cl2^{i}	0.93	2.80	3.677 (4)	157
C8—H8A···Cl1^{ii}	0.93	2.77	3.646 (3)	157
C15—H15A···Cl1^{iii}	0.93	2.76	3.649 (4)	159

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

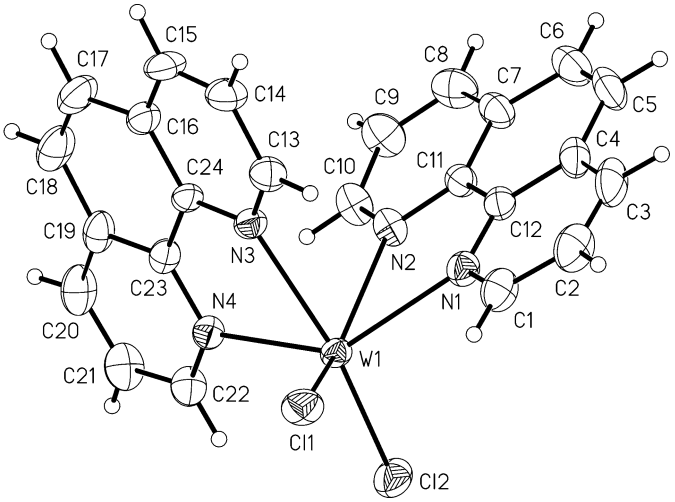


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 40% probability level.

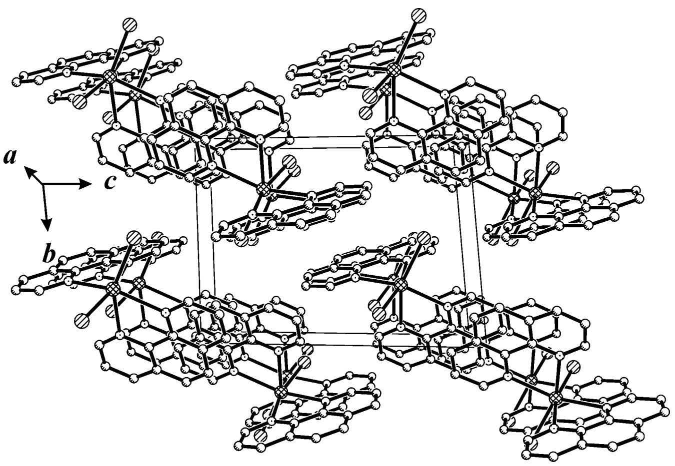


Figure 2
The crystal packing of the title complex, viewed pallel to (100). H atoms have been omitted.

H atoms were placed in geometrically calculated positions and refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). The deepest residual electron-density hole is located 0.64 Å from atom Cl1.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

This project was supported by the Expert Project of Key Basic Research of the Ministry of Science and Technology of China (grant No. 2003CCA00800), the Zhejiang Provincial Natural Science Foundation (grant No. Z203067), the Ningbo Municipal Natural Science Foundation (grant No. 2006 A610061) and the Scientific Research Found of Ningbo University (grant No. 0308037 and 2004624). The authors express sincere thank to Dr Y.-Q. Zheng for providing the study environment and for helpful comments.

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