electronic reprint

Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368
Editors: **W. Clegg and D. G. Watson**

Dichlorobis(phenothiazine-κS)palladium(II)

Xuanjun Zhang, Dan Li, Xiao-Ping Zhou and Seik Weng Ng

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dichlorobis(phenothiazine-κS)palladium(II)

Xuanjun Zhang,[a] Dan Li,[a]* Xiao-Ping Zhou[a] and Seik Weng Ng[b]

[a]Department of Chemistry, Shantou University, Shantou, Guangdong 515063, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: dli@stu.edu.cn

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.024
wR factor = 0.065
Data-to-parameter ratio = 17.8

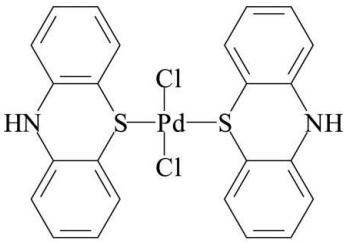
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 18 February 2005
Accepted 22 February 2005
Online 26 February 2005

In the title complex, $[PdCl_2(C_{12}H_9NS)_2]$, the Pd atom lies on a center of inversion and is coordinated by two chloride anions and two S atoms from two phenothiazine ligands, forming a square-planar $PdCl_2S_2$ geometry. In the crystal structure, a one-dimensional polymer structure is constructed *via* N—H···Cl and N—H···N hydrogen bonds.

Comment

Phenothiazine, its *N*-alkyl derivatives, and metal–phenothiazine complexes are biologically active compounds. Although many transition metal–phenothiazine complexes have been synthesized and characterized, reports of their crystal structures are relatively limited (Coe *et al.*, 1998; Kidd *et al.*, 1996; Kroener *et al.*, 1988; Zhang, Xie *et al.*, 2003; Zhang, Yu *et al.*, 2003). The consequent lack of structural information has hampered an understanding of the chemistry of this system. We report here the synthesis and crystal structure of a new phenothiazine derivative, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1 and selected bond distances and angles are given in Table 1. Atom Pd1 lies on a center of inversion and is coordinated by two chloride anions and two S atoms from two phenothiazine ligands. The bond angles about the Pd atom [exactly 180° for Cl—Pd—Cl and S—Pd—S, and 84.05 (2) and 95.95 (2)° for Cl—Pd—S] confirm that it is in a square-planar $PdCl_2S_2$ geometry. The Pd—S1 bond length [2.3378 (5) Å] lies within the normal range.

In the crystal structure, molecules of (I) are linked *via* N—H···Cl and N—H···N hydrogen bonds, forming one-dimensional chains extending in the **b** direction. Details of the hydrogen bonding are given in Table 2 and Fig. 2.

Experimental

The title compound, (I), was synthesized by self-assembly of phenothiazine and palladium chloride in acetonitrile in a 2:1 molar ratio. A phenothiazine solution was placed on one side of a fritted U-tube and on the other side $PdCl_2$ was added (approximately a stoi-

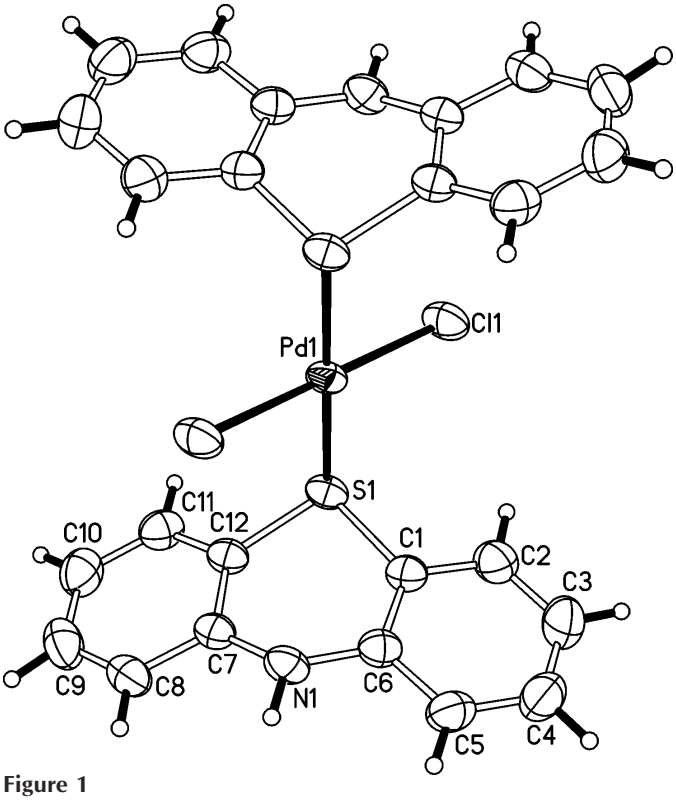


Figure 1
ORTEPII plot (Johnson, 1976) of compound (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level. Unlabeled atoms are related to labeled atoms by the symmetry operation $1-x, 1-y, 1-z$.

chiometric amount). CH_3CN was then added to equalize the hydrostatic pressures on both sides. After 5–6 d, well formed crystals suitable for X-ray analysis were obtained in the solutions on both sides of the frit.

Crystal data

$[PdCl_2(C_{12}H_9NS)_2]$	$Z = 1$
$M_r = 575.82$	$D_x = 1.699$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.6036$ (5) Å	Cell parameters from 3594 reflections
$b = 8.8540$ (5) Å	$\theta = 2.4$–$27.8°$
$c = 8.9240$ (5) Å	$\mu = 1.26$ mm^{-1}
$\alpha = 70.985$ (1)°	$T = 295$ (2) K
$\beta = 82.297$ (1)°	Block, brown
$\gamma = 86.860$ (1)°	0.25 × 0.24 × 0.20 mm
$V = 562.84$ (6) Å^3	

Data collection

Bruker SMART APEX area-detector diffractometer	2526 independent reflections
φ and ω scans	2416 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)	$R_{int} = 0.015$
$T_{min} = 0.659$, $T_{max} = 0.786$	$\theta_{max} = 27.5°$
4874 measured reflections	$h = -9 \rightarrow 9$
	$k = -11 \rightarrow 11$
	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.0826P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.024$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.065$	$\Delta\rho_{max} = 0.30$ e Å^{-3}
$S = 1.05$	$\Delta\rho_{min} = -0.50$ e Å^{-3}
2526 reflections	
142 parameters	
H-atom parameters constrained	

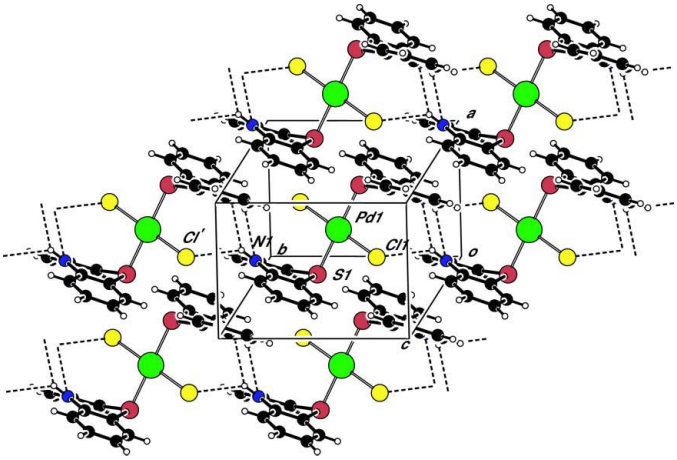


Figure 2
A view of the crystal packing of (I), showing the N—H···Cl and N—H···N hydrogen bonds (dashed lines).

Table 1
Selected geometric parameters (Å, °).

Pd1—Cl1	2.3091 (5)	Pd1—S1	2.3378 (5)
Cl1—Pd1—Cl1^{i}	180	Cl1—Pd1—S1^{i}	95.95 (2)
Cl1—Pd1—S1	84.05 (2)	S1—Pd1—S1^{i}	180

Symmetry code: (i) $1-x, 1-y, 1-z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1^{ii}	0.86	2.67	3.359 (2)	138
N1—H1···N1^{iii}	0.86	2.49	3.109 (2)	130

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

The H atoms were positioned geometrically (C—H = 0.93 Å and N—H = 0.86 Å) and were included in the refinement with $U_{iso}(H) = 1.2U_{eq}(C,N)$ in the riding-model approximation.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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*.

We thank the National Natural Science Foundation of China (Nos. 20271031 and 29901004), the Natural Science Foundation of Guangdong Province (No. 021240) and the University of Malaya for supporting this study.

References

Bruker (1999). *SADABS, SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Coe, B. J., Harris, J. A., Harrington, L. J., Jeffery, J. C., Rees, L. H., Houberchts, S. & Persoons, A. (1998). *Inorg. Chem.* **37**, 3391–3399.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kidd, S. E., Hambley, T. W., Hevér, A., Nelson, M. J. & Molnar, J. (1996). *J. Inorg. Biochem.* **62**, 171–181.
Kroener, R., Heeg, M. J. & Deutsch, E. (1988). *Inorg. Chem.* **27**, 558–566.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Zhang, X., Xie, Y., Yu, W., Zhao, Q., Jiang, M. & Tian, Y. (2003). *Inorg. Chem.* **42**, 3734–3737.
Zhang, X., Yu, W., Xie, Y., Zhao, Q. & Tian, Y. (2003). *Inorg. Chem. Commun.* **6**, 1338–1340.