

Xuanjun Zhang,<sup>a</sup> Dan Li,<sup>a\*</sup>  
Xiao-Ping Zhou<sup>a</sup> and  
Seik Weng Ng<sup>b</sup><sup>a</sup>Department of Chemistry, Shantou University,  
Shantou, Guangdong 515063, People's  
Republic of China, and <sup>b</sup>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  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.024  
 $wR$  factor = 0.065  
Data-to-parameter ratio = 17.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Dichlorobis(phenothiazine- $\kappa$ S)palladium(II)

In the title complex,  $[\text{PdCl}_2(\text{C}_{12}\text{H}_9\text{NS})_2]$ , 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  $\text{PdCl}_2\text{S}_2$  geometry. In the crystal structure, a one-dimensional polymer structure is constructed *via*  $\text{N}-\text{H}\cdots\text{Cl}$  and  $\text{N}-\text{H}\cdots\text{N}$  hydrogen bonds.

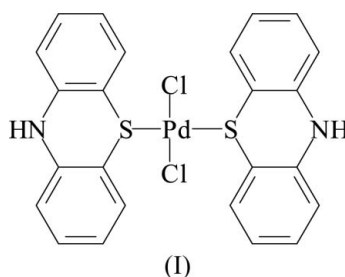
Received 18 February 2005

Accepted 22 February 2005

Online 26 February 2005

## 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).

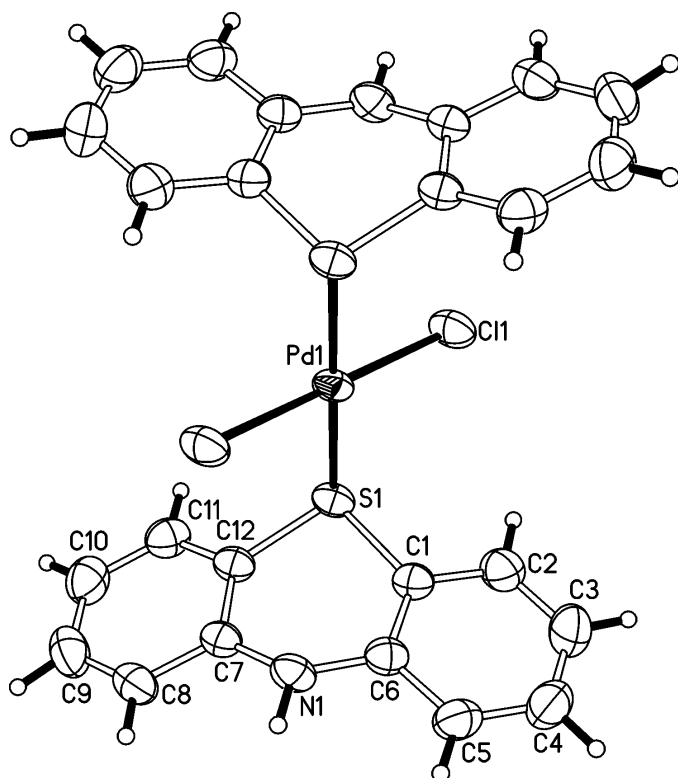


The molecular structure of (I) is illustrated in Fig. 1 and selected bond distances and angles are given in Table 1. Atom Pd1 lies on a center of inversion and is coordinated by two chloride anions and two S atoms from two phenothiazine ligands. The bond angles about the Pd atom [exactly  $180^\circ$  for  $\text{Cl}-\text{Pd}-\text{Cl}$  and  $\text{S}-\text{Pd}-\text{S}$ , and  $84.05(2)$  and  $95.95(2)^\circ$  for  $\text{Cl}-\text{Pd}-\text{S}$ ] confirm that it is in a square-planar  $\text{PdCl}_2\text{S}_2$  geometry. The  $\text{Pd}-\text{S}1$  bond length [ $2.3378(5)$  Å] lies within the normal range.

In the crystal structure, molecules of (I) are linked *via*  $\text{N}-\text{H}\cdots\text{Cl}$  and  $\text{N}-\text{H}\cdots\text{N}$  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  $\text{PdCl}_2$  was added (approximately a stoi-



**Figure 1**  
ORTEP 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).  $\text{CH}_3\text{CN}$  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

$[\text{PdCl}_2(\text{C}_{12}\text{H}_9\text{NS})_2]$   
 $M_r = 575.82$   
 Triclinic,  $P\bar{1}$   
 $a = 7.6036(5) \text{ \AA}$   
 $b = 8.8540(5) \text{ \AA}$   
 $c = 8.9240(5) \text{ \AA}$   
 $\alpha = 70.985(1)^\circ$   
 $\beta = 82.297(1)^\circ$   
 $\gamma = 86.860(1)^\circ$   
 $V = 562.84(6) \text{ \AA}^3$

$Z = 1$   
 $D_x = 1.699 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 3594 reflections  
 $\theta = 2.4\text{--}27.8^\circ$   
 $\mu = 1.26 \text{ mm}^{-1}$   
 $T = 295(2) \text{ K}$   
 Block, brown  
 $0.25 \times 0.24 \times 0.20 \text{ mm}$

#### Data collection

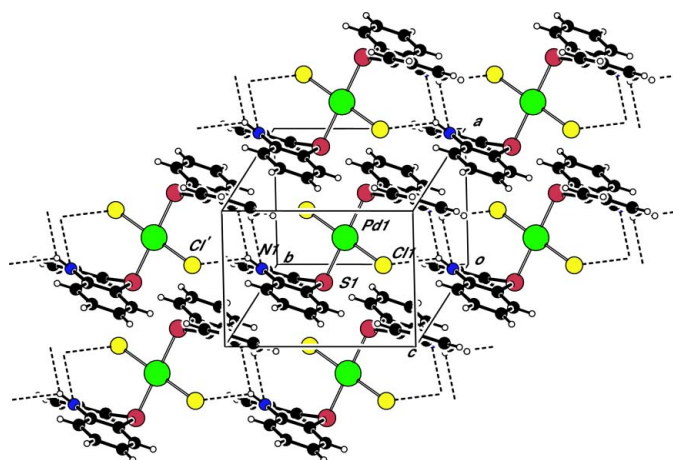
Bruker SMART APEX area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 1999)  
 $T_{\min} = 0.659, T_{\max} = 0.786$   
 4874 measured reflections

2526 independent reflections  
 2416 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.015$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = -9 \rightarrow 9$   
 $k = -11 \rightarrow 11$   
 $l = -11 \rightarrow 11$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.024$   
 $wR(F^2) = 0.065$   
 $S = 1.05$   
 2526 reflections  
 142 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.0826P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.50 \text{ e \AA}^{-3}$



**Figure 2**  
A view of the crystal packing of (I), showing the  $\text{N-H}\cdots\text{Cl}$  and  $\text{N-H}\cdots\text{N}$  hydrogen bonds (dashed lines).

**Table 1**

Selected geometric parameters ( $\text{\AA}, ^\circ$ ).

Pd1—Cl1	2.3091 (5)	Pd1—S1	2.3378 (5)
Cl1—Pd1—Cl1 <sup>i</sup>	180	Cl1—Pd1—S1 <sup>i</sup>	95.95 (2)
Cl1—Pd1—S1	84.05 (2)	S1—Pd1—S1 <sup>i</sup>	180

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

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}, ^\circ$ ).

$D\text{—H}\cdots A$	$D\text{—H}$	$\text{H}\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
$\text{N1—H1}\cdots\text{Cl1}^{\text{ii}}$	0.86	2.67	3.359 (2)	138
$\text{N1—H1}\cdots\text{N1}^{\text{iii}}$	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 ( $\text{C—H} = 0.93 \text{ \AA}$  and  $\text{N—H} = 0.86 \text{ \AA}$ ) and were included in the refinement with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$  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: ORTEP II (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). ORTEP II. 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.