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Bis(piperidine-1-dithiocarbamato-$\kappa^2 S,S'$)palladium(II)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.021
wR factor = 0.055
Data-to-parameter ratio = 15.9

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

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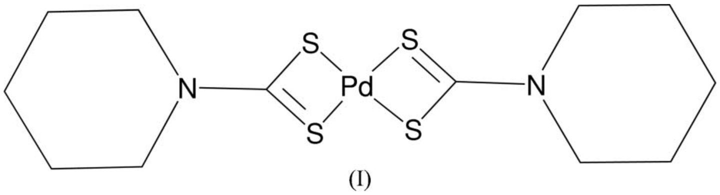
In the title centrosymmetric complex, [Pd($C_6H_{10}NS_2$)$_2$], the Pd^{II} atom has a square-planar environment with two dithiocarbamate ligands coordinating through their S atoms in a bidentate chelating fashion.

Comment

The interest in Pd^{II} complexes containing *N*,*S* donors has recently been increased with the aim of synthesizing antitumor drugs with reduced toxicity compared to cisplatin and its analogs. The dithiocarbamato complexes, [*M*(S_2CNEt_2)(*L*)]NO_3 where *M* is Pt or Pd and *L* is 2,2′-bipyridyl or 1, 10-phenanthroline, show antitumor activity against leukemic cells (Mital *et al.*, 1989). Sulfur-containing compounds show bactericidal and antifungal activity. The dithiocarbamate group has also received much attention due to its ability to act as a bidentate ligand. Dithiocarbamates can easily undergo many substitution reactions in cation-bound ligands or between the ligands themselves. The introduction of the dithiocarbamate group in α-amino acids lead to molecules with up to three potential bonding residues, *viz.* amino (–NH_2, –NH*R*, –NH*RR*′), dithiocarbamate (–C*SS*′) and carboxylate (–COO–) (Ronconi *et al.*, 2005). The dithiocabamate derivatives of α-amino acids of aliphatic, cyclic and aromatic amines such as [Pd(ethylsarcosinedithiocarbamate)(*n*-propylamine)Cl] and [Pd(ethylsarcosinedithiocarbamate)(cyclobutylamine)Cl] are able to conjugate their antineoplastic activity with a low nephrotoxicity (Alvedi *et al.*, 2004). Dithiocarbamates such as sodium diethyldithiocarbamate and ammonium pyrrolidinedithiocarbamate have been widely used as chelating agents for the determination of trace metals due to their ability to form thermodynamically stable metal complexes (Dapaah & Ayame, 1998). The title compound, (I), was obtained from a dithiocarbamate derived from piperidine.



(I)

The asymmetric unit contains one-half of the [Pd($C_6H_{10}NS_2$)$_2$] complex, the other half being generated by a crystallographic inversion centre; atom Pd1 lies on the inversion centre (Fig. 1). The Pd^{II} atom has a square-planar environment with the two dithiocarbamate ligands coordinating through their sulfur atoms in a bidentate chelating fashion. The Pd—S bond lengths (Table 1) show normal values (Orpen *et al.*, 1989). The piperidine ring of the ligand adopts a chair conformation. No significant hydrogen-bonding interactions are observed in the crystal structure except for weak intramolecular C—H···S interactions (Table 2) which generate rings of graph-set motif *S*(5) (Bernstein *et al.*, 1995).

Experimental

Compound (I) was synthesized by the addition of piperidine-1-dithiocarboxylato-*S*;*SS′*(Garje & Jain, 2003), (0.54 g, 3.38 mmol) to a suspension of $PdCl_2$ (0.3 g, 1.69 mmol, Aldrich) in CH_2Cl_2 (20 ml). The resulting mixture was refluxed for 1 h, giving a clear bright-yellow solution. The solution was evaporated under reduced pressure to give a yellow solid, which was recrystallized from dichloromethane/*n*-hexane (9:1) to afford compound (I) (0.66 g, yield: 70%)

Crystal data

$[Pd(C_6H_{10}NS_2)_2]$
$M_r = 426.94$
Monoclinic, $P2_1/c$
$a = 6.1094$ (10) Å
$b = 8.5797$ (15) Å
$c = 15.339$ (3) Å
$\beta = 95.197$ (3)°
$V = 800.7$ (2) Å^3
$Z = 2$

$D_x = 1.771$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3118 reflections
$\theta = 2.7–25.0°$
$\mu = 1.67$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.19 × 0.09 × 0.06 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.742$, $T_{max} = 0.907$
3891 measured reflections

1399 independent reflections
1215 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 25.0°$
$h = -7 \rightarrow 7$
$k = -9 \rightarrow 10$
$l = -18 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.055$
$S = 1.18$
1399 reflections
88 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.027P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—S1	2.3189 (7)	Pd1—S2	2.3300 (7)
$S1^i$—Pd1—S1	180	S1—Pd1—$S2^i$	104.48 (2)
S1—Pd1—S2	75.52 (2)	S2—Pd1—$S2^i$	180

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

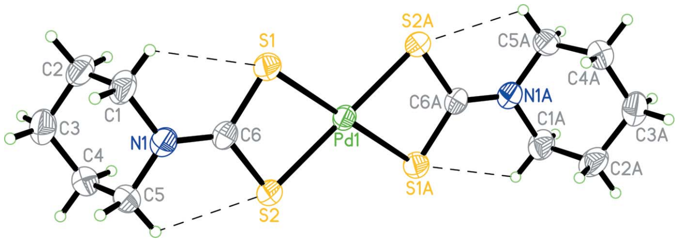


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Atoms labeled with the suffix A are generated by the symmetry operation ($-x$, $-y$, $-z$). Dashed lines indicate C—H···S interactions.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···S1	0.97	2.57	3.085 (3)	113
C5—H5*B*···S2	0.97	2.60	3.111 (3)	113

All H atoms were positioned geometrically and allowed to ride on the parent atoms, with C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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