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Tetrakis[1-*tert*-butyl-1*H*-imidazole-2(3*H*)-thione-κ*S*]-palladium(II) dichloride dichloromethane disolvate

Yu-Ling Wang,* Wen-Hua Bi, Xing Li and Rong Cao

State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, People's Republic of China

Correspondence e-mail: wyl76@ms.fjirsm.ac.cn

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In the title compound, $[Pd(C_7H_{12}N_2)_4]Cl_2 \cdot 2CH_2Cl_2$, the Pd atom lies on an inversion center and has a square-planar coordination involving the S atoms of the four 1-*tert*-butylimidazoline-2(3*H*)-thione ligands. The two chloride ions are not involved in coordination, but are engaged in hydrogen bonding.

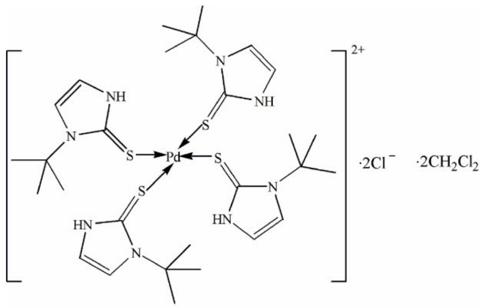
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.044
wR factor = 0.106
Data-to-parameter ratio = 17.6

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

Comment

Heterocyclic mercaptans form complexes with transition metals with potential applications in analytical chemistry and the pharmaceutical industry (Hussain *et al.*, 1990). The ability of 1-alkylimidazoline-2(3*H*)thione to coordinate to palladium(II) in aqueous solution at different pH levels has been demonstrated previously (Butler *et al.*, 1983). The present study details the structure of the 1-*tert*-butylimidazoline-2(3*H*)-thione complex of palladium dichloride, which exists as a bis(dichloromethane) solvate. The title compound, (I), consists of a square-planar-coordinated palladium(II) cation, chloride anions and dichloromethane molecules (Fig. 1). The structure is similar to that of the methyl-substituted derivative, which crystallizes as a dihydrate (Butler *et al.*, 1983). The Pd atom is located at a center of inversion and has a square-planar coordination involving four S atoms. Coordination of the ligand to Pd leads to a lengthening of the exocyclic C—S distance from 1.700 (2) (White *et al.*, 2003) to 1.723 (4) and 1.709 (3) Å, but a shortening of the endocyclic C—N bond from 1.348 (2) to 1.336 (5) and 1.328 (4) Å. The Cl^- anions are located above and below the PdS_4 plane, 3.9048 (11) Å from the Pd atom. The ligand participates in N—H···Cl hydrogen bonding with the anion (Table 2 and Fig. 2).



(I)

Experimental

1-*tert*-Butylimidazoline-2(3*H*)-thione (0.063 g, 0.4 mmol), which was prepared according to the procedure of Cassidy *et al.* (1999), and $Pd(CH_3CN)_2Cl_2$ (0.026 g, 0.1 mmol) were stirred in dichloromethane (10 ml) overnight. The solution was filtered; red crystals were

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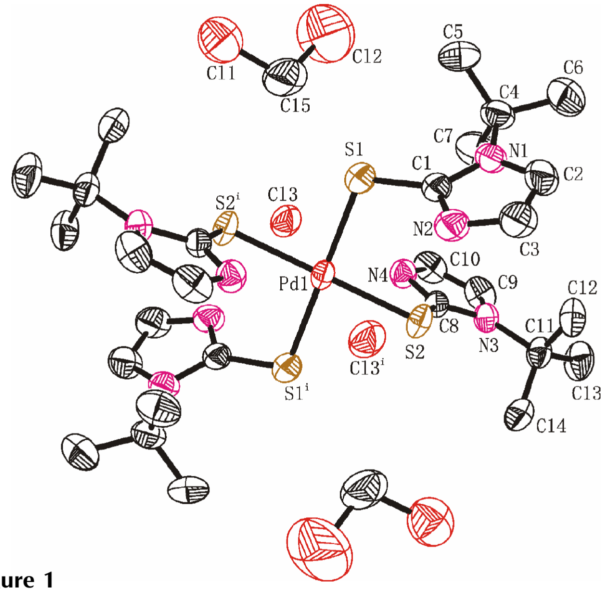

Figure 1
The title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

obtained (yield ~70%) after a week by the slow diffusion of hexane into the filtrate.

Crystal data

$[Pd(C_7H_{12}N_2)_4]Cl_2 \cdot 2CH_2Cl_2$
$M_r = 972.14$
Monoclinic, $P2_1/c$
$a = 9.3724$ (2) Å
$b = 21.9527$ (4) Å
$c = 11.4474$ (2) Å
$\beta = 111.493$ (1)°
$V = 2191.51$ (7) Å^3
$Z = 2$

$D_x = 1.473$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7773 reflections
$\theta = 1.9$–25.2°
$\mu = 1.01$ mm^{-1}
$T = 293$ (2) K
Block, red
0.48 × 0.32 × 0.10 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.685$, $T_{max} = 0.904$
11 352 measured reflections

3927 independent reflections
3378 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.2°$
$h = -10 \rightarrow 11$
$k = -16 \rightarrow 26$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.106$
$S = 1.00$
3927 reflections
223 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 4.8056P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.97$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—S1	2.3193 (11)	S1—C1	1.723 (4)
Pd1—S2	2.3263 (9)	N4—C8	1.328 (4)
S2—C8	1.709 (3)		
S1—Pd1—S2^i	84.68 (4)	C8—S2—Pd1	102.76 (12)
S1—Pd1—S2	95.32 (4)	C1—S1—Pd1	109.04 (13)

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

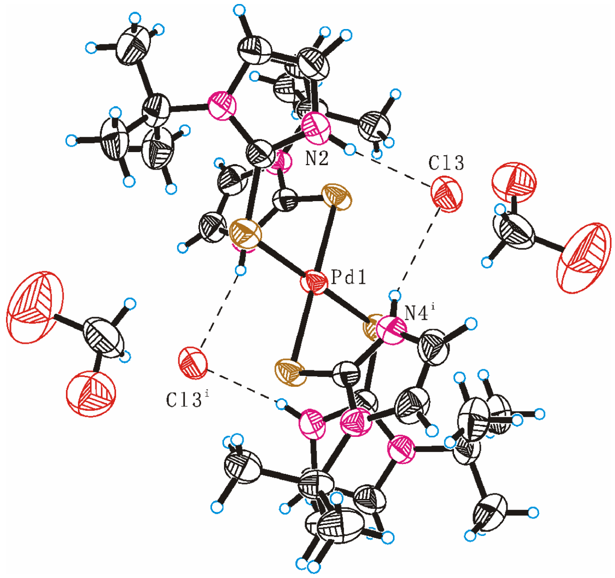

Figure 2
A view of the N—H···Cl hydrogen bonds.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···Cl3	0.86	2.24	3.080 (4)	165
N4—H4*A*···Cl3^i	0.86	2.40	3.131 (3)	143

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

All H atoms were placed in calculated positions and treated using a riding-model approximation (C—H = 0.93 Å for aromatic H atoms, 0.97 Å for methyl H atoms and 0.96 Å for methylene H atoms, and N—H = 0.86 Å). The U_{iso}(H) values were set at $1.2U_{eq}$ of the parent atoms (1.5 for the methyl H atoms).

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*.

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