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Bis(tetra-*n*-butylammonium) bis(1,2-dicyanoethylene-1,2-dithiolato-κ^2S,S')palladate(II)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
R factor = 0.065
wR factor = 0.148
Data-to-parameter ratio = 18.0

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

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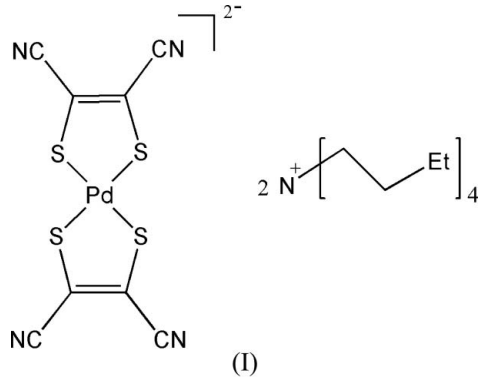
In the title compound, $(C_{16}H_{36}N_2)_2[Pd(C_4N_2S_2)_2]$, the Pd^{II} ion, located on an inversion centre, is coordinated by the four S atoms of the two ligands, giving a coordination geometry very close to ideal square-planar.

Comment

Transition metal–thiolate complexes have been studied extensively because of their potential applications. Sulfur-containing transition metal complexes are also well known as catalysts for many reactions. In addition, S-containing metalloproteins, such as blue copper proteins, play an important role in biological systems and are crucial to the development of transition-metal–thiolate chemistry (Ferguson-Miller & Babcock, 1996). Within this field, there has been an increasing interest in the structural and physical properties of complexes including 1,2-dicyanoethylene-1,2-dithiolate and corresponding anions as ligands (Waters *et al.*, 2006). We report here the crystal structure of one such complex, (I).



(I)

In (I), the Pd^{II} ion is located on an inversion centre and is coordinated by the four S atoms of the two ligands. Pd—S bond lengths [2.2945 (15) and 2.2947 (16) Å] are in good agreement with those found in related compounds containing the same anion (e.g. Connelly *et al.*, 1992). Bond angles around Pd^{II} are 180 and 90.10 (6)°; the Pd^{II} ion in (I) therefore has a coordination environment very close to ideal square-planar (Fig. 1).

Experimental

The disodium salt of 1,2-dicyanoethylene-1,2-dithiolate was prepared following a reported procedure (Simmons *et al.*, 1962). A solution of 3.9 g of this starting material in 12 ml of a 1:1 water–ethanol mixture was warmed on a steam bath. $Pd(PhCN)_2Cl_2$ (3.8 g in 10 ml of ethanol) was added dropwise, with stirring. The resulting green solution was filtered. To the filtrate was added a solution of NBu_4Br

(2.6 g) in ethanol (5 ml). After cooling, an olive-green precipitate of (I) was obtained by filtration. Well-shaped green crystals suitable for X-ray diffraction analysis were grown by slow diffusion of diethyl ether into an acetonitrile solution of (I).

Crystal data

$(C_{16}H_{36}N_2)_2[Pd(C_4N_2S_2)_2]$	$\gamma = 65.476\ (2)^\circ$
$M_r = 871.74$	$V = 1191.6\ (2)$ Å^3
Triclinic, $P\overline{1}$	$Z = 1$
$a = 9.7986\ (11)$ Å	Mo $K\alpha$ radiation
$b = 10.8084\ (12)$ Å	$\mu = 0.60$ mm^{-1}
$c = 12.4030\ (13)$ Å	$T = 293\ (2)$ K
$\alpha = 85.631\ (2)^\circ$	$0.56 \times 0.26 \times 0.22$ mm
$\beta = 87.964\ (2)^\circ$	

Data collection

Siemens SMART CCD diffractometer	6213 measured reflections 4181 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	3178 reflections with $I > 2\sigma(I)$ $R_{int} = 0.031$
$T_{min} = 0.713$, $T_{max} = 0.877$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$	232 parameters
$wR(F^2) = 0.148$	H-atom parameters constrained
$S = 1.15$	$\Delta\rho_{max} = 0.47$ e Å^{-3}
4181 reflections	$\Delta\rho_{min} = -0.64$ e Å^{-3}

H atoms were placed in calculated positions and refined as riding, with C—H = 0.97 (methylene CH_2) or 0.96 Å (methyl CH_3), and $U_{iso}(H) = 1.5U_{eq}$(methyl C) or $1.2U_{eq}$(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1994); data reduction: *XPREP* (Siemens, 1994); program(s) used to solve structure: *SHELXTL* (Siemens, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

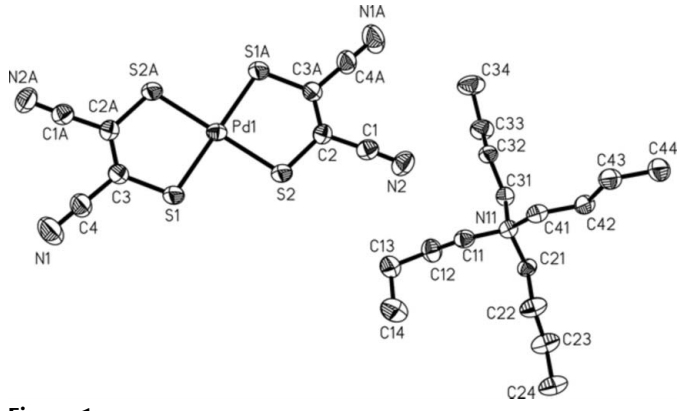


Figure 1
The asymmetric unit of (I), extended to show the complete coordination of Pd, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted for clarity. [Symmetry code: (A) $3 - x, -2 - y, 3 - z$.]

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