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cis-[1,2-Bis(propylsulfinyl)ethane-S,S']-dichloroplatinum(II)

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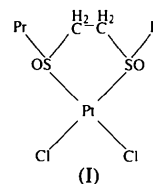
Abstract

The coordination geometry about Pt in the title complex, [PtCl$_2$(C$_8$H$_{18}$O$_2$S$_2$)], deviates slightly from square planar. The Pt ion is bonded to two Cl atoms, arranged in a *cis* configuration, and two S atoms of the bidentate disulfoxide ligand.

Comment

The title complex, (I), was synthesized as part of a research program on the organometallic chemistry of

tin and the behaviour of these compounds towards Pt and Pd complexes. The synthesis was carried out in H$_2$O/MeOH solution, with a yield of 85%, as follows:

K$_2$PtCl$_4$ + (PrSOCH$_2$)$_2$ →
2KCl + *cis*-[PtCl$_2$(PrSOCH$_2$)$_2$] (I)
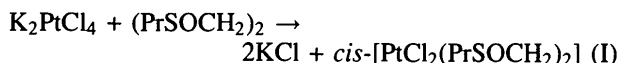
The average Pt—Cl bond length of 2.317 Å is in the normal range, according to the compilation of Orpen *et al.* (1989), which gives a value of 2.323 (38) Å for 293 examples of four-coordinate Pt. The disulfoxide ligand is bonded to the Pt atom at two coordination sites through the S atoms. The average Pt—S distance of 2.209 Å is not significantly different from the values found in *cis*-[PtCl$_2$(*meso*-PhSOCH$_2$CH$_2$SOPh)] [2.217 (2) and 2.209 (2) Å] and *cis*-[PtCl$_2$(*rac*-PhSOCH$_2$CH$_2$SOPh)] [2.192 (4) and 2.188 (4) Å] (Cattalini, Michelon, Marangoni & Pelizzi, 1979), and *cis*-[PtCl$_2$(*rac-cis*-PhSO-
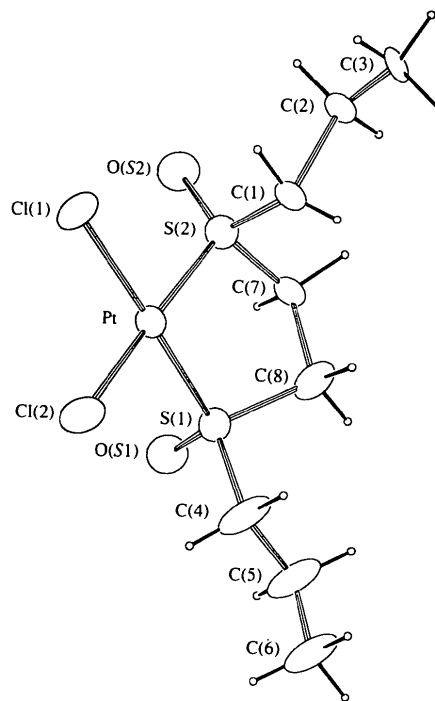

Fig. 1. A perspective view of the title molecule with atom numbering. Displacement ellipsoids are drawn at the 50% probability level.
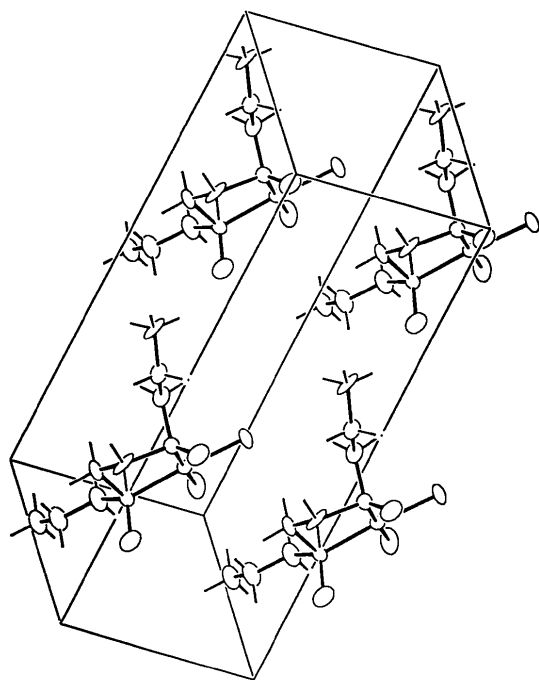
Fig. 2. A packing diagram of the title compound.

$V = 1396.1$ (4) Å³	Rectangular plate
$Z = 4$	$0.60 \times 0.43 \times 0.15$ mm
$D_x = 2.27$ Mg m⁻³	Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.04$
	$\theta_{max} = 25°$
ω–2θ scans	$h = 0 \rightarrow 8$
Absorption correction:	$k = 0 \rightarrow 11$
refined from ΔF	$l = 0 \rightarrow 22$
(*DIFABS*; Walker & Stuart, 1983)	2 standard reflections frequency: 30 min
1464 measured reflections	intensity decay: not significant
1437 independent reflections	
1369 observed reflections	
$[I > 3\sigma(I)]$	

Refinement

Refinement on F	$w = 2.4089/[\sigma^2(F_o)$
$R = 0.047$	$+ 0.00071F_o^2]$
$wR = 0.052$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.43$	$\Delta\rho_{max} = 1.31$ e Å⁻³
1369 reflections	$\Delta\rho_{min} = -3.83$ e Å⁻³
136 parameters	Atomic scattering factors
H-atom parameters not refined	from Cromer & Mann (1968)

CH=CHSOPh)] [2.195 (5) and 2.203 (5) Å] (Filgueiras, Holland, Johnson & Raithby, 1982). The molecular structure of (I) is shown in Fig. 1, together with the atom labeling. The packing of the molecules in the unit cell is shown in Fig. 2.

The distances of the Pt, S and Cl atoms from the least-squares plane defined by the Pt coordination shell in *cis*-[PtCl₂(*rac-cis*-PrSOCH₂CH₂SOPr)] and *cis*-[PtCl₂(*rac-cis*-PhSOCH=CHSOPh)] (Filgueiras, Holland, Johnson & Raithby, 1982), *cis*-[PtCl₂(*rac*-PhSOCH₂CH₂SOPh)] and *cis*-[PtCl₂(*meso*-PhSOCH₂CH₂SOPh)] (Cattalini, Michelon, Marangoni & Pelizzi, 1979), and [PtCl₂-(C₄H₈OS)₂] (Melanson, de la Chevrotière & Rochon, 1985) are given in Table 3. In the title complex, the square-planar coordination is slightly distorted [the maximum distance from the least-squares plane is 0.097 Å for atom S(1)].

Experimental

Crystal data

[PtCl₂(C₈H₁₈O₂S₂)]	Mo $K\alpha$ radiation
$M_r = 476.34$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 25
$P2_12_12_1$	reflections
$a = 7.360$ (2) Å	$\theta = 13.45$–$22.68°$
$b = 9.793$ (2) Å	$\mu = 10.81$ mm⁻¹
$c = 19.369$ (2) Å	$T = 296$ K

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}a_i.a_j.$$

	x	y	z	B_{eq}
Pt	0.1098 (1)	0.5153 (1)	0.4903 (1)	2.34 (2)
Cl(1)	−0.1032 (6)	0.5203 (6)	0.4031 (3)	3.7 (2)
Cl(2)	−0.0734 (7)	0.6548 (6)	0.5583 (3)	4.0 (2)
S(1)	0.3089 (6)	0.4866 (5)	0.5741 (2)	2.7 (1)
S(2)	0.2911 (6)	0.3955 (5)	0.4222 (2)	2.7 (1)
O(S1)	0.271 (2)	0.366 (1)	0.6159 (7)	3.8 (4)
O(S2)	0.222 (2)	0.273 (2)	0.3890 (8)	4.7 (5)
C(1)	0.375 (3)	0.514 (2)	0.359 (1)	3.5 (6)
C(2)	0.487 (2)	0.437 (2)	0.3033 (9)	3.5 (6)
C(3)	0.555 (3)	0.531 (4)	0.250 (1)	6 (1)
C(4)	0.352 (3)	0.631 (2)	0.630 (1)	3.6 (6)
C(5)	0.497 (3)	0.605 (3)	0.682 (1)	5.0 (8)
C(6)	0.511 (5)	0.717 (3)	0.731 (1)	7 (1)
C(7)	0.491 (2)	0.362 (2)	0.4718 (9)	3.0 (5)
C(8)	0.522 (2)	0.472 (2)	0.529 (1)	4.2 (6)

Table 2. *Selected geometric parameters* (Å, °)

Pt—Cl(1)	2.305 (5)	S(2)—C(1)	1.80 (2)
Pt—Cl(2)	2.328 (5)	S(2)—C(7)	1.79 (2)
Pt—S(1)	2.205 (4)	C(1)—C(2)	1.55 (3)
Pt—S(2)	2.213 (4)	C(2)—C(3)	1.47 (4)
S(1)—O(S1)	1.46 (1)	C(4)—C(5)	1.49 (3)
S(1)—C(4)	1.81 (2)	C(5)—C(6)	1.45 (4)
S(1)—C(8)	1.80 (2)	C(7)—C(8)	1.58 (3)
S(2)—O(S2)	1.45 (2)		
Cl(1)—Pt—Cl(2)	90.5 (2)	O(S2)—S(2)—C(1)	110.6 (9)
Cl(1)—Pt—S(1)	173.8 (2)	O(S2)—S(2)—C(7)	112.0 (9)
Cl(1)—Pt—S(2)	89.1 (2)	C(1)—S(2)—C(7)	101.6 (8)
Cl(2)—Pt—S(1)	92.5 (2)	S(2)—C(1)—C(2)	110 (1)
Cl(2)—Pt—S(2)	176.1 (2)	C(1)—C(2)—C(3)	111 (1)
S(1)—Pt—S(2)	88.3 (2)	S(1)—C(4)—C(5)	113 (1)
O(S1)—S(1)—C(4)	109.5 (8)	C(4)—C(5)—C(6)	111 (1)
O(S1)—S(1)—C(8)	112.4 (9)	S(2)—C(7)—C(8)	112 (1)
C(4)—S(1)—C(8)	101.0 (9)	S(1)—C(8)—C(7)	105 (1)

Table 3. *Deviations (Å) of atoms from the least-squares plane defined by* Pt, S(1), S(2), Cl(1) *and* Cl(2) *for five related complexes*

Complexes: (a) *cis*-[PtCl$_2$(*rac-cis*-PrSOCH$_2$CH$_2$SOPr)] (this work), (b) *cis*-[PtCl$_2$(*rac-cis*-PhSOCH=CHSOPh)] (Filgueiras, Holland, Johnson & Raithby, 1982), (c) *cis*-[PtCl$_2$(*rac*-PhSOCH$_2$CH$_2$SOPh)] (Cattalini, Michelon, Marangoni & Pelizzi, 1979), (d) *cis*-[PtCl$_2$(*meso*-PhSOCH$_2$CH$_2$SOPh)] (Cattalini, Michelon, Marangoni & Pelizzi, 1979) and (e) [PtCl$_2$(C$_4$H$_8$OS)$_2$] (Melanson, de la Chevrotière & Rochon, 1985).

	(a)	(b)	(c)	(d)	(e)
Pt	0.012 (1)	0.012	0.011	0.000	0.001
S(1)	−0.097 (4)	−0.008	−0.005	0.130	0.022
S(2)	0.094 (4)	0.002	−0.001	−0.130	0.001
Cl(1)	−0.094 (5)	−0.008	−0.001	−0.121	0.001
Cl(2)	0.085 (5)	0.002	−0.005	0.121	0.030

The structure was solved by the Patterson method and successive Fourier electron density difference maps, and refined by full-matrix least-squares procedures with anisotropic temperature factors for all non-H atoms. The positions of all H atoms were generated geometrically in the final stages of refinement. Anomalous-dispersion corrections were taken from Cromer & Liberman (1970) for non-H atoms and from Stewart, Davidson & Simpson (1965) for H atoms. Programs used were *SDP* (Enraf–Nonius, 1985), *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: BK1039). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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An Aminobenzyldioxocyclam Nickel(II) Complex

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Abstract

The title compound, [6-(4-aminobenzyl)-1,11-diaza-4,8-diazanidacyclotetradecane-5,7-dione(2−)-$\kappa^4 N^{1,4,8,11}$]-nickel(II), [Ni(C$_{17}$H$_{25}$N$_5$O$_2$)], has mirror symmetry with Ni—N(amide) and Ni—N(amine) bond distances of 1.862 (5) and 1.925 (5) Å, respectively. The aromatic ring of the side chain is tipped 21.3 (3)° towards the plane containing the Ni and four N atoms. The distance from the Ni atom to the center of the aromatic ring is 3.551 (5) Å.

Comment

Polyazamacrocyclic ligands have many applications in chemistry, biology and medicine. We have been examining the use of carbon-substituted polyazamacrocycles as bifunctional chelating agents (BCAs) (Moi, Meares, McCall, Cole & DeNardo, 1985). We report here the X-ray crystal structure of an aminobenzyldioxocyclam nickel complex, (I).

(I)
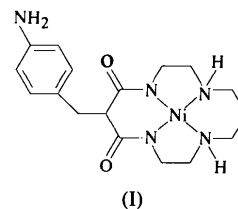
The most striking feature of the aminobenzyldioxocyclam nickel structure is that the aromatic ring of the side chain sits 3.551 (5) Å above the Ni atom, with N(amide)—Ni—*Ctr* (where *Ctr* is the center of the aromatic ring) and N(amine)—Ni—*Ctr* angles of 86.3 (1) and 96.3 (1)°, respectively. The aromatic ring is nearly parallel to the plane containing the four N atoms, making a dihedral angle of 21.3 (3)° with it. This is quite different from the structure observed in the previously reported phenyldioxocyclam nickel complex, where the phenyl ring is perpendicular to the plane of the four N atoms with an H atom directed towards the Ni atom (Chinn, Busch & Alcock, 1993).

The structure displays the expected square-planar geometry about the nickel(II) atom coordinated by