

C2	0.3724 (10)	-0.2172 (21)	0.2982 (12)	2.4 (4)†
C3	0.3672 (11)	-0.3706 (24)	0.2633 (13)	3.8 (5)†
C4	0.4257 (11)	-0.4625 (24)	0.2717 (12)	4.0 (5)†
C5	0.4887 (11)	-0.3948 (24)	0.3096 (13)	4.1 (5)†
C6	0.4948 (11)	-0.2470 (25)	0.3407 (13)	4.1 (5)†
C7	0.4367 (10)	-0.1464 (20)	0.3350 (11)	2.1 (4)†
C8	0.4418 (9)	0.0137 (25)	0.3700 (10)	2.9 (4)†
C9	0.5061 (10)	0.0827 (23)	0.4081 (12)	3.3 (5)†
C10	0.5094 (11)	0.2332 (25)	0.4430 (13)	3.9 (5)†
C11	0.4513 (12)	0.3158 (24)	0.4473 (13)	4.3 (5)†
C12	0.3877 (1)	0.2544 (23)	0.4097 (13)	3.5 (5)†
C13	0.3827 (10)	0.1080 (22)	0.3708 (11)	2.7 (4)†
C14	0.3157 (9)	0.0406 (18)	0.3278 (10)	1.8 (4)†
N2	0.2577 (8)	0.1106 (15)	0.3209 (9)	2.2 (3)†
N3	0.1624 (8)	-0.0998 (18)	0.3759 (10)	0.0395 (64)
C15	0.1335 (12)	0.0259 (31)	0.4193 (13)	0.0664 (76)
C16	0.0792 (12)	0.1100 (30)	0.3621 (16)	0.0688 (83)
N4	0.1033 (9)	0.1587 (17)	0.2833 (10)	0.0452 (56)
N5	0.0936 (8)	-0.1423 (17)	0.1895 (10)	0.0408 (48)
C17	0.0958 (12)	-0.1354 (22)	0.0970 (14)	0.0551 (96)
C18	0.1145 (12)	0.0368 (24)	0.0819 (12)	0.0536 (79)
N6	0.1759 (8)	0.0854 (18)	0.1470 (10)	0.0395 (45)
Br1	0.9452 (1)	0.0854 (3)	0.1552 (2)	0.0592 (7)
Br2	0.1764 (1)	0.0171 (3)	0.6748 (2)	0.0657 (8)
Br3	0.3205 (1)	-0.0022 (3)	0.0807 (1)	0.0625 (7)
O1	0.6990 (8)	0.1547 (16)	0.5224 (9)	0.0733 (53)
O2	0.2205 (8)	0.0961 (17)	0.8964 (9)	0.0740 (50)
O3	0.3095 (9)	0.1075 (18)	0.5829 (10)	0.0963 (57)

† Isotropic displacement parameter, *B*.

Table 2. Bond distances (Å) and angles (°)

Rh—N1	1.996 (15)	C7—C8	1.45 (3)
Rh—N2	2.007 (14)	C8—C9	1.41 (3)
Rh—N3	2.066 (15)	C8—C13	1.42 (3)
Rh—N4	2.058 (16)	C9—C10	1.38 (3)
Rh—N5	2.065 (16)	C10—C11	1.37 (3)
Rh—N6	2.029 (16)	C11—C12	1.37 (3)
N1—C1	1.29 (2)	C12—C13	1.37 (3)
C1—C2	1.46 (3)	C13—C14	1.47 (3)
C1—C14	1.45 (2)	C14—N2	1.28 (2)
C2—C3	1.40 (3)	N3—C15	1.46 (3)
C2—C7	1.41 (3)	C15—C16	1.44 (3)
C3—C4	1.38 (3)	C16—N4	1.52 (3)
C4—C5	1.39 (3)	N5—C17	1.51 (3)
C5—C6	1.33 (3)	C17—C18	1.52 (3)
C6—C7	1.42 (3)	C18—N6	1.48 (3)
N1—Rh—N2	77.1 (6)	C7—C2—C3	122.0 (17)
N1—Rh—N3	92.2 (6)	C4—C3—C2	119.5 (19)
N1—Rh—N4	172.0 (6)	C5—C4—C3	118.4 (19)
N1—Rh—N5	96.5 (6)	C6—C5—C4	122.7 (20)
N1—Rh—N6	91.2 (6)	C7—C6—C5	121.8 (20)
N2—Rh—N3	90.2 (6)	C6—C7—C2	115.3 (17)
N2—Rh—N4	96.3 (6)	C8—C7—C6	121.8 (17)
N2—Rh—N5	172.1 (6)	C8—C7—C2	122.8 (17)
N2—Rh—N6	92.6 (6)	C9—C8—C7	121.6 (17)
N3—Rh—N4	83.3 (6)	C13—C8—C7	122.3 (17)
N3—Rh—N5	94.6 (6)	C13—C8—C9	116.1 (17)
N3—Rh—N6	176.0 (6)	C10—C9—C8	120.4 (18)
N4—Rh—N5	90.5 (6)	C11—C10—C9	121.7 (20)
N4—Rh—N6	93.6 (6)	C12—C11—C10	119.7 (20)
N5—Rh—N6	82.9 (6)	C13—C12—C11	119.9 (19)
Rh—N1—C1	117.8 (12)	C12—C13—C8	122.0 (18)
Rh—N2—C14	117.4 (12)	C14—C13—C8	116.6 (16)
Rh—N3—C15	107.5 (13)	C14—C13—C12	121.4 (17)
Rh—N4—C16	108.8 (13)	C13—C14—C1	120.8 (15)
Rh—N5—C17	110.0 (12)	N2—C14—C1	114.4 (15)
Rh—N6—C18	110.5 (12)	N2—C14—C13	124.8 (15)
C2—C1—N1	125.8 (16)	C16—C15—N3	111.4 (19)
C14—C1—N1	113.3 (15)	N4—C16—C15	109.5 (19)
C14—C1—C2	120.9 (15)	C18—C17—N5	105.1 (16)
C3—C2—C1	120.5 (17)	N6—C18—C17	109.8 (17)
C7—C2—C1	117.3 (16)		

R values quoted are on *F*; the high value for all data with $F_o^2 > 0$ is due to the small size of the crystal and the consequently weak data. There were essentially no reflections beyond 20°

in θ . H atoms were positioned by calculation (C—H or N—H, 0.95 Å) and were repositioned once near the end of refinement. $w = 1/\sigma^2(F_o^2)$, where $\sigma^2(F_o^2)$ for a single datum is derived from counting statistics plus an additional term, $(0.014I)^2$. Variances of the merged data were obtained by propagation of error plus another additional term, $(0.014I)^2$. Values for f' were taken from Cromer (1974). Programs from the *CRYM* crystallographic computing system (Duchamp, 1964) were used together with *ORTEP* (Johnson, 1976).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55249 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1011]

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Structure of *trans*-Dichloro(dimethyl sulfoxide)(piperidine)platinum(II)

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Abstract

In the title compound the Pt atom coordinates one S atom, one N atom and two chloride ions in the *trans* position. The coordination geometry is square planar with distances Pt—S 2.212 (6), Pt—N 2.083 (18), and Pt—Cl 2.292 (6) and 2.296 (5) Å.

Comment

The crystal structure of $[\text{Pt}(\text{dmsso})(\text{piperidine})\text{Cl}_2]$ [dmsso = dimethyl sulfoxide, $(\text{CH}_3)_2\text{SO}$] was determined as part of a systematic study of *cis/trans* influences in square-planar platinum(II) compounds (Bugarcic, Norén, Oskarsson, Stålhandske & Elding, 1991; Boström, Strandberg, Norén & Oskarsson, 1991). The compound was prepared by mixing a solution of 0.2 g $\text{K}[\text{Pt}(\text{dmsso})\text{Cl}_3]$ (Kukushkin, Vyaz'menskii & Zorina, 1968) in 10 ml of water with 1 ml of a solution containing 0.5 ml piperidine/10 ml water (molar ratio Pt/piperidine 1:1.05). The beaker was covered with parafilm. After a few weeks, yellow-orange crystals and a white precipitate were formed. A single yellow-orange crystal was selected for use in further investigations.

The $[\text{Pt}(\text{dmsso})(\text{piperidine})\text{Cl}_2]$ molecules are probably linked by weak hydrogen bonds between the dimethyl sulfoxide O atom and the piperidine N atom (Table 2). The S atom in the dimethyl sulfoxide, the piperidine N atom and the two chloride ions are bonded to the Pt atom in a distorted square-planar *trans* arrangement. Deviations from the least-squares plane through these atoms are Pt 0.0024 (7), S -0.007 (6), N -0.007 (17), Cl(1) 0.006 (6), Cl(2) 0.006 (6) Å. The shortest Pt—Pt distance is 6.871 (2) Å. The molecular structure and atomic

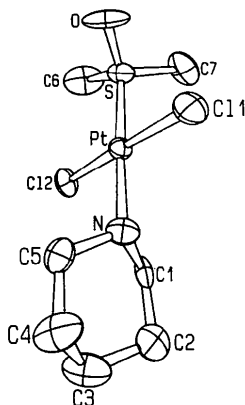


Fig. 1. Atomic numbering in $[\text{Pt}(\text{dmsso})(\text{piperidine})\text{Cl}_2]$.

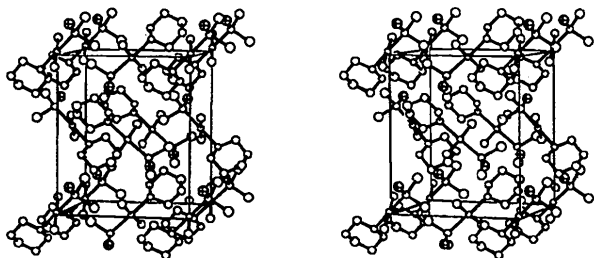


Fig. 2. Crystal packing in $[\text{Pt}(\text{dmsso})(\text{piperidine})\text{Cl}_2]$ viewed along the *a* axis. The O atom is marked.

numbering are shown in Fig. 1 and a packing diagram of the unit-cell contents is in Fig. 2. Selected interatomic distances and angles for the complex are given in Table 2; as expected, the angles around the Pt atom are close to 90 and 180°.

The S atom in the dimethyl sulfoxide is in an approximately tetrahedral environment with angles ranging from 101 to 118° (Table 2). The Pt—S distance of 2.212 (6) Å is within the range (2.21–2.23 Å) observed in other compounds (Table 3) which have an N atom *trans* and two Cl atoms *cis* relative to S. The *trans* influences of the different types of N atom on the Pt—S bond length (Table 3) are therefore about the same. The Pt—Cl distances are in good agreement both with those found in this type of compound (Table 3) and with an average value [2.304 (7) Å] calculated from 12 crystal structures with $[\text{PtCl}_4]^{2-}$ (Bengtsson & Oskarsson, 1992).

The least-squares plane through the piperidine ring is nearly perpendicular [$96(1)^\circ$] to the coordination plane. As expected, the angles in the ring are close to tetrahedral (106–113°; Table 2). The Pt—N distance is the longest observed in any of the *trans*- $[\text{Pt}(\text{dmsso})(\text{N-binding})\text{Cl}_2]$ compounds in Table 3 but unfortunately the errors involved are too large to

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

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Pt	0.08970 (7)	0.01621 (7)	0.02408 (6)	0.0345 (2)
Cl(1)	0.30032 (55)	0.04603 (68)	-0.05982 (54)	0.0606 (21)
Cl(2)	-0.12342 (52)	-0.02119 (58)	0.10275 (46)	0.0512 (16)
S	0.14804 (59)	0.16610 (56)	0.13994 (51)	0.0463 (18)
N	0.0431 (17)	-0.1224 (16)	-0.0895 (16)	0.040 (5)
O	0.2800 (19)	0.1486 (21)	0.2002 (17)	0.079 (8)
C(1)	-0.0841 (23)	-0.0947 (19)	-0.1533 (17)	0.042 (6)
C(2)	-0.1086 (24)	-0.1915 (25)	-0.2450 (21)	0.056 (8)
C(3)	-0.1144 (27)	-0.3279 (29)	-0.1910 (25)	0.071 (11)
C(4)	0.0204 (29)	-0.3587 (26)	-0.1278 (23)	0.065 (9)
C(5)	0.0375 (27)	-0.2577 (20)	-0.0417 (18)	0.051 (8)
C(6)	0.0093 (36)	0.2004 (34)	0.2353 (29)	0.080 (12)
C(7)	0.1561 (33)	0.3204 (22)	0.0645 (29)	0.077 (11)

Table 2. Geometric parameters (Å, °)

Pt—S	2.212 (6)	Pt—Cl(1)	2.292 (6)
Pt—N	2.083 (18)	Pt—Cl(2)	2.296 (5)
N—C(1)	1.48 (3)	N—C(5)	1.54 (3)
C(1)—C(2)	1.55 (3)	C(2)—C(3)	1.58 (4)
C(3)—C(4)	1.55 (4)	C(4)—C(5)	1.52 (4)
S—O	1.48 (2)	S—C(6)	1.82 (4)
S—C(7)	1.88 (3)		
O...N'	3.14(3)		
S—Pt—N	177.2 (5)	Cl(1)—Pt—Cl(2)	177.4 (2)
S—Pt—Cl(1)	89.1 (2)	S—Pt—Cl(2)	93.6 (2)
N—Pt—Cl(1)	88.2 (5)	N—Pt—Cl(2)	89.2 (5)
Pt—N—C(1)	114 (1)	Pt—N—C(5)	113 (1)
C(1)—N—C(5)	111 (2)	N—C(1)—C(2)	113 (2)
C(1)—C(2)—C(3)	106 (2)	C(2)—C(3)—C(4)	112 (2)
C(3)—C(4)—C(5)	108 (3)	C(4)—C(5)—N	112 (2)
Pt—S—C(6)	113 (1)	Pt—S—O	118 (1)
Pt—S—C(7)	107 (1)	O—S—C(6)	108 (1)
O—S—C(7)	109 (1)	C(6)—S—C(7)	101 (2)

Table 3. Comparison of similar *trans*-[Pt(dmsO)(N-binding)Cl₂] compounds

Compound	Pt—S (Å)	Pt—N (Å)	Pt—Cl (Å)	Reference
<i>trans</i> -[Pt(dmsO)(cytidine)Cl ₂]	2.220 (4)	2.034 (13)	2.291 (5), 2.310 (4)	Melanson & Rochon (1978a)
<i>trans</i> -[Pt(dmsO)(2-picoline)Cl ₂]	2.212 (3)	2.046 (10)	2.282 (3), 2.295 (4)	Melanson & Rochon (1978b)
<i>trans</i> -[Pt(dmsO)(NH ₃)Cl ₂]	2.209 (1)	2.053 (4)	2.296 (1), 2.306 (1)	Viossat, Khodadad & Rodier (1991)
<i>trans</i> -[Pt(dmsO)(pyridine)Cl ₂]	2.224 (2)	2.052 (6)	2.299 (3), 2.276 (3)	
	2.225 (3)	2.061 (10)	2.305 (2), 2.294 (3)	Caruso, Spagna & Zambonelli (1980)
<i>trans</i> -[Pt(dmsO)(isopropylamine)Cl ₂]	2.220 (2)	2.067 (6)	2.315 (2), 2.290 (2)	Melanson & Rochon (1984)
<i>trans</i> -[Pt(dmsO)(piperidine)Cl ₂]	2.212 (6)	2.083 (18)	2.292 (6), 2.296 (5)	This work

allow any firm conclusions to be drawn about the *trans* influence of S on the piperidine N atom compared to that on other types of N atom.

Experimental

Crystal data



$M_r = 429.26$

Orthorhombic

$P2_12_12_1$

$a = 9.551 (2) \text{ \AA}$

$b = 10.486 (3) \text{ \AA}$

$c = 12.543 (2) \text{ \AA}$

$V = 1256.2 (3) \text{ \AA}^3$

$Z = 4$

$D_x = 2.273 \text{ Mg m}^{-3}$

Data collection

CAD-4 diffractometer

$\omega/2\theta$ scans

Absorption correction:

by integration from crystal shape

$T_{\min} = 0.1986$, $T_{\max} = 0.4152$

1746 measured reflections

1746 independent reflections

1329 observed reflections

$[I > 3\sigma(I)]$

Refinement

Refinement on F^2

Final $R = 0.048$

$wR = 0.061$

$S = 1.04$

1329 reflections

119 parameters

H-atom parameters not refined

$w = (\sigma^2/4|F_o|^2 + 0.038|F_o|^2 + 3.0)^{-1}$

$(\Delta/\sigma)_{\max} = 0.076$

The structure was solved by standard Patterson and difference Fourier methods and refined by full-matrix least-squares calculations employing programs amended by Lundgren (1982). The final coordinates are given in Table 1. The H atoms could not be located in difference Fourier maps and were not included in the structure-factor calculations.

Financial support from the Swedish Natural Science Research Council is gratefully acknowledged.

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 4.9\text{--}15.5^\circ$

$\mu = 11.86 \text{ mm}^{-1}$

$T = 295\text{--}296 \text{ K}$

Prism

$0.22 \times 0.18 \times 0.05 \text{ mm}$

Yellow-orange

$R_{\text{int}} = 0.0068$ (based on standard reflections)

$\theta_{\max} = 28^\circ$

$h = 0 \rightarrow 12$

$k = -13 \rightarrow 0$

$l = 0 \rightarrow 16$

3 standard reflections

frequency: 120 min

intensity variation:

$< \pm 2\%$

$\Delta\rho_{\max} = 1.46 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -1.32 \text{ e \AA}^{-3}$

Extinction correction: isotropic

Extinction coefficient: 370

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55293 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1016]

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Regioselective Pyrazine Ring Reduction of 4-(Quinoxalin-2-yl)-1,3-dithiole-2-thione

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(Received 27 February 1992; accepted 5 June 1992)

Abstract

The crystal structure of 4-(4-benzoyloxycarbonyl-1,2,3,4-tetrahydroquinoxalin-2-yl)-1,3-dithiole-2-thione [benzyl 1,2,3,4-tetrahydro-3-(2-thioxo-1,3-dithiol-4-yl)quinoxaline-1-carboxylate] shows that