

***trans*-Dichloro(dimethyl sulphoxide)(pyridine)platinum(II)**

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Abstract. $C_7H_{11}Cl_2NOPtS$, triclinic, $P\bar{1}$, $a = 8.677$ (2), $b = 10.710$ (3), $c = 13.358$ (3) Å, $\alpha = 85.54$ (2), $\beta = 89.25$ (2), $\gamma = 69.17$ (2)°, $V = 1156.5$ (5) Å³, $Z = 4$, $D_m = 2.44$ (1), $D_x = 2.431$ Mg m⁻³, $\mu(\text{Mo } K\alpha) = 12.86$ mm⁻¹. For 3789 counter data, final $R = 0.038$. The two molecules in the asymmetric unit have almost identical bond lengths and angles but different conformations.

Introduction. Yellow crystals of the title compound were obtained from a solution of *trans*-[Pt(ethylene)(pyridine)Cl₂] in dimethyl sulphoxide, Me₂SO, prepared at room temperature.

Intensities were collected from a crystal $0.30 \times 0.25 \times 0.10$ mm on a Syntex $P2_1$ computer-controlled diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. 5597 independent reflections were measured up to $2\theta = 56^\circ$ by the ω -scan technique at a variable speed (2 to 29.3° min⁻¹).

The intensity data were processed as previously described (Bachechi, Zambonelli & Marcotrigiano, 1977), with an uncertainty factor $p = 0.017$ calculated from the variance of the standard reflections (McCandlish, Stout & Andrews, 1975). The values of I and $\sigma(I)$ were corrected for Lorentz, polarization and shape-

anisotropy (Spagna & Zambonelli, 1971) effects. 3789 independent reflections having $I > 3\sigma(I)$ were used in the subsequent calculations.

The structure was solved by the heavy-atom method and refined by least squares to a final R of 0.038 ($R_w = 0.047$). The fixed contribution of the H atoms, geometrically positioned, was included in the last cycles, during which the non-hydrogen atoms were allowed to vibrate anisotropically. The refinement was carried out with a two-blocks approximation of the normal-equation matrix. The function minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = 4F_o^2/\sigma^2(F_o^2)$.

Scattering factors and anomalous-dispersion terms were taken from *International Tables for X-ray Crystallography* (1974). The calculations were performed with local programs on the Univac 1100/20 computer of the University of Rome and on the HP 21MX minicomputer of the CNR Research Area.

The final atomic coordinates are given in Table 1.* Bond distances and angles are reported in Table 2.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending division as Supplementary Publication No. SUP 34909 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates of the two independent molecules*

E.s.d.'s are given in parentheses.

	Molecule (I)			Molecule (II)		
	<i>x</i>	<i>y</i>	<i>z</i>	<i>x</i>	<i>y</i>	<i>z</i>
Pt	0.2058 (0)	0.3204 (0)	0.3785 (0)	0.4928 (0)	0.1536 (0)	0.1056 (0)
Cl(1)	-0.0302 (2)	0.4946 (2)	0.3256 (2)	0.6412 (3)	0.2925 (3)	0.0721 (2)
Cl(2)	0.4432 (3)	0.1582 (3)	0.4382 (2)	0.3634 (3)	0.0022 (3)	0.1399 (2)
S	0.0665 (2)	0.1814 (2)	0.3843 (2)	0.2657 (3)	0.3112 (3)	0.0400 (2)
O	-0.0534 (8)	0.1989 (7)	0.4645 (6)	0.1082 (7)	0.2889 (8)	0.0469 (6)
N	0.3307 (7)	0.4513 (7)	0.3745 (5)	0.7079 (8)	0.0086 (8)	0.1615 (5)
C(1)	0.2812 (9)	0.5601 (8)	0.4265 (7)	0.7159 (12)	-0.0463 (11)	0.2571 (8)
C(2)	0.3676 (10)	0.6474 (10)	0.4254 (7)	0.8581 (14)	-0.1447 (11)	0.2939 (8)
C(3)	0.5038 (11)	0.6231 (10)	0.3668 (8)	0.9934 (14)	-0.1900 (11)	0.2344 (10)
C(4)	0.5527 (10)	0.5133 (12)	0.3094 (8)	0.9841 (11)	-0.1365 (12)	0.1379 (10)
C(5)	0.4656 (10)	0.4281 (10)	0.3139 (7)	0.8384 (11)	-0.0358 (10)	0.1010 (8)
C(6)	0.1964 (11)	0.0079 (9)	0.3933 (8)	0.3048 (13)	0.3435 (12)	-0.0888 (8)
C(7)	-0.0333 (11)	0.1928 (11)	0.2670 (8)	0.2320 (11)	0.4671 (10)	0.0864 (8)

Table 2. Bond lengths (Å) and angles (°) in the two independent molecules with e.s.d.'s in parentheses

	(I)	(II)
Pt—Cl(1)	2.299 (3)	2.305 (2)
Pt—Cl(2)	2.276 (3)	2.294 (3)
Pt—S	2.224 (2)	2.225 (3)
Pt—N	2.052 (6)	2.061 (10)
S—O	1.458 (8)	1.469 (7)
S—C(6)	1.793 (12)	1.781 (11)
S—C(7)	1.774 (11)	1.751 (12)
N—C(1)	1.338 (12)	1.356 (13)
C(1)—C(2)	1.391 (12)	1.373 (20)
C(2)—C(3)	1.366 (14)	1.369 (19)
C(3)—C(4)	1.389 (17)	1.361 (19)
C(4)—C(5)	1.375 (13)	1.403 (19)
C(5)—N	1.373 (11)	1.347 (14)
Cl(1)—Pt—Cl(2)	175.8 (1)	175.7 (1)
Cl(1)—Pt—S	89.7 (1)	90.8 (1)
Cl(1)—Pt—N	89.6 (3)	87.8 (2)
Cl(2)—Pt—S	93.4 (1)	93.3 (1)
Cl(2)—Pt—N	87.3 (3)	88.1 (2)
S—Pt—N	178.9 (2)	177.8 (2)
Pt—S—O	115.4 (3)	119.3 (4)
Pt—S—C(6)	113.5 (3)	107.3 (5)
Pt—S—C(7)	110.1 (4)	112.0 (4)
O—S—C(6)	107.1 (5)	108.6 (5)
O—S—C(7)	109.4 (5)	106.7 (5)
C(6)—S—C(7)	100.3 (6)	101.5 (6)
Pt—N—C(1)	121.8 (5)	120.3 (8)
Pt—N—C(5)	118.7 (6)	119.6 (7)
C(1)—N—C(5)	119.5 (7)	120.0 (10)
N—C(1)—C(2)	121.9 (8)	120.4 (11)
C(1)—C(2)—C(3)	118.7 (10)	120.7 (11)
C(2)—C(3)—C(4)	119.9 (9)	118.8 (13)
C(3)—C(4)—C(5)	119.6 (9)	120.2 (12)
C(4)—C(5)—N	120.4 (10)	119.9 (10)

Discussion. Fig. 1 shows a perspective view of the two molecules in the asymmetric unit, and Fig. 2 illustrates the packing.

The two independent molecules (I and II) have almost identical bond lengths and angles which com-

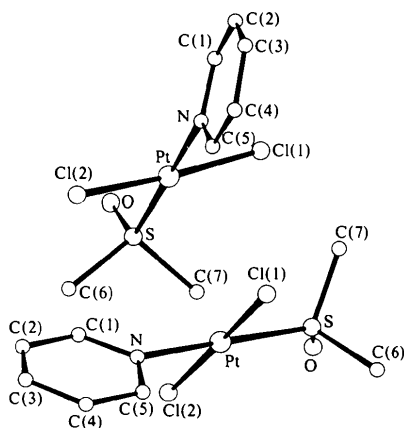


Fig. 1. The two independent molecules, (I) and (II), shown in a perspective view of the asymmetric unit.

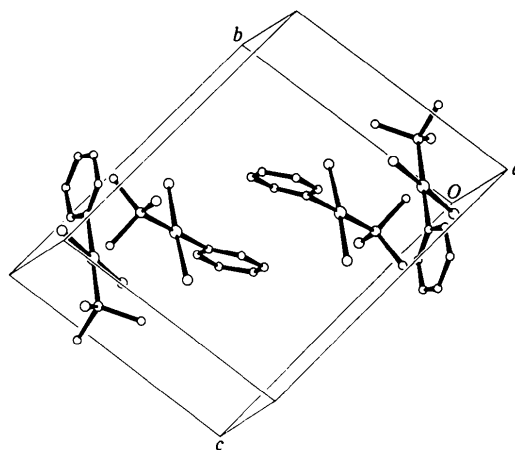


Fig. 2. Packing of the molecules. None of the contact distances are less than the sums of the van der Waals radii.

pare well with those in the analogous *trans*-[Pt(2-picoline)(Me₂SO)Cl₂] (Melanson & Rochon, 1978). However, they have different conformations.

In both molecules Pt has the expected square-planar coordination. The four donor atoms, two Cl which are *trans* to each other, the S atom of the Me₂SO molecule and the N atom of the pyridine molecule, are almost exactly coplanar with the metal. The largest angular deviation from the regular values involves the Cl—Pt—Cl angle. The two Cl atoms bend away from the bulky Me₂SO group, closing the Cl—Pt—Cl angle to 175.8 (1) in (I) and 175.7 (1)° in (II).

The Pt—Cl lengths in the two independent molecules average 2.294 (2) Å, a value which is normal for mutually *trans* Pt—Cl bonds.

The Pt—S bonds as well as the Pt—N bonds are equivalent in the two molecules. They average 2.225 (2) and 2.057 (8) Å respectively.

The geometry of the coordinated Me₂SO, approximately tetrahedral, is similar in the two molecules. Averaged bond lengths and angles are: S—O = 1.464 (7), S—C = 1.775 (11) Å; Pt—S—O = 117.4 (3), Pt—S—C = 110.7 (4), O—S—C = 118.0 (5), C—S—C = 100.9 (6)°. They are in fair agreement with those in uncoordinated Me₂SO (Viswamitra & Kannan, 1966; Thomas, Shoemaker & Eriks, 1966).

The bond lengths within the planar pyridine rings in (I) and (II) average 1.354 (12) and 1.378 (15) Å for N—C and C—C respectively, and the angles are close to 120°.

The only difference between (I) and (II) concerns the relative orientation of the pyridine rings and the Me₂SO groups with respect to the PtCl₂ moiety. The angle between the coordination plane and the pyridine ring is 59.4 in (I) and 120.6° in (II), and the dihedral angle Cl(1)—Pt—S—O is -70.4 in (I) and -174.0° in (II).

The two molecules (I) and (II) appear faced and the two coordination planes are roughly parallel (dihedral

angle = 9.6°), one molecule being rotated in the coordination plane by ca 90° with respect to the other. However, there are no intermolecular contacts less than the sums of the van der Waals radii.

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Bis(oxamide oximato)platinum(II)–Hydrogen Chloride (1:2), Form (II)

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Abstract. $C_4H_{10}N_8O_4Pt \cdot 2HCl$, $[Pt(C_2H_5N_4O_2)_2] \cdot 2HCl$, $M_r = 502.17$, $P\bar{1}$, $a = 6.787$ (2), $b = 8.876$ (4), $c = 12.486$ (6) Å, $\alpha = 112.58$ (3), $\beta = 81.67$ (3), $\gamma = 112.71$ (2)°, $Z = 2$, $d_c = 2.60$ Mg m⁻³; final $R = 0.067$ for 4065 reflections. A H-bonded intramolecular O–Cl–O bridge exists instead of an O–H–O bridge between oxime O atoms.

Introduction. In preceding papers we have dealt with the structural variability of oxamide oxime (diaminoglyoxime, oaoH₂) complexes of Ni and Pt. References are given in Endres & Schlicksupp (1979), where we describe how $[Pt(oaoH)_2] \cdot 2HCl$ crystallizes simultaneously in two different modifications from HCl solution, and where we report the structure of form (I). The structure of form (II) is the subject of this communication.

A crystal 0.06 × 0.17 × 0.19 mm was used for the investigation. Rotation and Weissenberg photographs showed the crystal to be triclinic and gave an estimate for the lattice constants. Exact lattice parameters were calculated by least squares (Berdesinski & Nuber, 1966) from the θ values of 59 reflections centred on a diffractometer. Data collection on a computer-controlled diffractometer (Siemens AED, Mo $K\alpha$ radiation, θ – 2θ scans, five-value method, $4 < 2\theta < 66^\circ$) yielded 4065 observed independent reflections with $I > 3.0\sigma(I)$. They were corrected for Lorentz and polarization factors. An absorption correction by numerical integration was applied.

The Pt position was derived from a Patterson synthesis, which favoured the choice of the centrosymmetric space group. Fourier syntheses showed the positions of the other atoms. Refinement by full-matrix least squares with anisotropic temperature factors converged with $R = 0.067$ (maximum shift/error 0.009). A final difference map showed no maxima

Table 1. Positional parameters ($\times 10^4$) and isotropic temperature factors equivalent to the refined anisotropic values

	x	y	z	U (Å ²)
Pt	2426 (1)	4837 (1)	2039 (1)	0.034
Cl(1)	3949 (7)	8205 (5)	7888 (3)	0.054
Cl(2)	1963 (6)	1911 (4)	6195 (3)	0.045
O(1)	2076 (19)	2480 (12)	3362 (9)	0.052
O(2)	2645 (18)	8531 (11)	3307 (8)	0.048
O(3)	2673 (18)	7279 (12)	822 (8)	0.049
O(4)	2223 (19)	1108 (11)	615 (8)	0.049
N(1)	2301 (21)	4111 (12)	3363 (9)	0.041
N(2)	2478 (19)	6979 (13)	3397 (9)	0.040
N(3)	2633 (19)	5633 (12)	738 (9)	0.038
N(4)	2415 (21)	2720 (13)	634 (9)	0.042
N(5)	2404 (21)	4993 (14)	5391 (9)	0.045
N(6)	2857 (23)	8372 (15)	5407 (9)	0.049
N(7)	2436 (21)	4912 (16)	–1264 (9)	0.046
N(8)	2165 (24)	1481 (14)	–1377 (9)	0.050
C(1)	2483 (22)	5269 (16)	4419 (10)	0.040
C(2)	2636 (26)	7029 (16)	4432 (10)	0.042
C(3)	2429 (22)	4525 (16)	–323 (10)	0.038
C(4)	2309 (25)	2783 (19)	–389 (11)	0.045