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Structure of *cis*-Dichlorobis(dimethyl sulfide)platinum(II)

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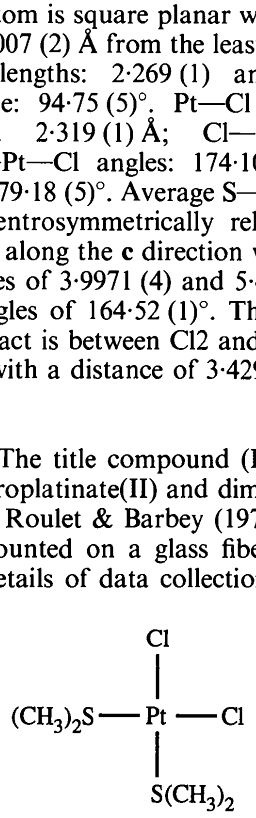
Abstract. $[PtCl_2\{S(CH_3)_2\}_2]$, $M_r = 390{\cdot}26$, monoclinic, $P2_1/n$, $a = 8{\cdot}719\,(2)$, $b = 13{\cdot}186\,(4)$, $c = 9{\cdot}328\,(1)$ Å, $\beta = 106{\cdot}30\,(1)°$, $V = 1029{\cdot}3\,(7)$ Å^3, $Z = 4$, $D_x = 2{\cdot}518$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 146{\cdot}2$ cm^{-1}, $F(000) = 720$, $T = 296\,(1)$ K, 3559 unique reflections measured, final $R = 0{\cdot}034$ over 2495 reflections having $F_o > 3{\cdot}0\sigma(F_o)$. The geometry about the Pt atom is square planar with a maximum deviation of 0·007 (2) Å from the least-squares plane. Pt—S bond lengths: 2·269 (1) and 2·272 (1) Å; S—Pt—S angle: 94·75 (5)°. Pt—Cl bond lengths: 2·315 (1) and 2·319 (1) Å; Cl—Pt—Cl angle: 89·69 (5)°. S—Pt—Cl angles: 174·10 (5), 91·11 (5), 84·44 (5) and 179·18 (5)°. Average S—C bond length: 1·785 (3) Å. Centrosymmetrically related molecules stack in chains along the **c** direction with alternating Pt—Pt distances of 3·9971 (4) and 5·4147 (4) Å, and Pt—Pt—Pt angles of 164·52 (1)°. The closest intermolecular contact is between Cl2 and Cl at $-x - \frac{1}{2}$, $y - \frac{1}{2}$, $-z - \frac{1}{2}$ with a distance of 3·429 (7) Å between them.

Experimental. The title compound (I) was prepared from tetrachloroplatinate(II) and dimethyl sulfide by the method of Roulet & Barbey (1973). The sample crystal was mounted on a glass fiber in a random orientation. Details of data collection and structure refinement are given in Table 1. Space group determined from systematic absences: $h0l$ with $h + l$ odd; $0k0$ with k odd. The position of the Pt atom was determined from a Patterson map, and the remaining atoms were located in succeeding difference Fourier syntheses. Refinement by full-matrix least squares with Enraf–Nonius *SDP/VAX* (Frenz, 1978); non-H atoms anisotropic; H atoms located by ΔF synthesis and placed in calculated positions with B_{iso} of the calculated H atoms given values of 1·3 times B_{eq} of the C atom. H atoms included in the structure-factor calculations riding on



$$\begin{array}{c} Cl \\ | \\ (CH_3)_2S-Pt-Cl \\ | \\ S(CH_3)_2 \end{array}$$

(I)

Table 1. *Experimental details*

Crystal description	Yellow, fragment of a plate, 0·38 × 0·30 × 0·30 mm
Instrument	Enraf–Nonius CAD-4 diffractometer, graphite monochromator
Corrections	Lorentz–polarization Linear decay (0·952–1·130 on I) Empirical absorption (0·38–0·99 on I) Extinction [3·23 (7) × 10^{-7}]
Maximum 2θ (°)	64·0
hkl ranges	h = 0–13 k = 0–19 l = − 13–13
No. of reflections	3894 total 3559 unique
R_{int} of averged reflections	0·031
No. unobserved reflections	1064
Reflections included	2495 with $F_o > 3\sigma(F_o)$
Solution	Heavy-atom method
Function minimized	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$
Weights	$4F_o^2\text{Lp}^2/[S^2(C + R^2B) + (0{\cdot}02F_o^2)^2]$ Lp = Lorentz–polarization S = scan rate C = total integrated peak count R = ratio of scan time to background counting time B = total background count
Parameters refined	83
Unweighted residual, R	0·034
Weighted residual, wR	0·038
Goodness of fit, S	1·62
Maximum Δ/σ	0·01

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Table 2. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Pt1	0·02468 (2)	0·48251 (2)	0·29481 (3)	2·619 (4)
Cl1	0·2822 (2)	0·4730 (1)	0·2728 (3)	4·93 (4)
Cl2	0·0302 (2)	0·3097 (1)	0·3421 (2)	3·94 (4)
S1	−0·2271 (2)	0·4742 (1)	0·3183 (2)	3·15 (3)
S2	0·0155 (2)	0·6518 (1)	0·2482 (2)	3·60 (3)
C1	−0·3238 (7)	0·5945 (6)	0·303 (1)	4·8 (2)
C2	−0·3433 (8)	0·4184 (7)	0·149 (1)	5·3 (2)
C3	0·1894 (8)	0·7079 (6)	0·3707 (9)	4·5 (2)
C4	0·062 (1)	0·6691 (7)	0·0741 (9)	5·8 (2)

Table 3. *Bond distances* (Å), *bond angles* (°) *and torsion angles* (°)

Pt1	Cl1	2·315 (1)	S1	C1	1·783 (6)
Pt1	Cl2	2·319 (1)	S1	C2	1·779 (7)
Pt1	S1	2·269 (1)	S2	C3	1·783 (6)
Pt1	S2	2·272 (1)	S2	C4	1·794 (6)

Cl1	Pt1	Cl2	89·69 (5)	Pt1	S1	C1	113·5 (2)
Cl1	Pt1	S1	174·10 (5)	Pt1	S1	C2	105·1 (2)
Cl1	Pt1	S2	91·11 (5)	C1	S1	C2	99·1 (3)
Cl2	Pt1	S1	84·44 (5)	Pt1	S2	C3	107·9 (2)
Cl2	Pt1	S2	179·18 (5)	Pt1	S2	C4	107·1 (3)
S1	Pt1	S2	94·75 (5)	C3	S2	C4	99·3 (3)

Cl2	Pt1	S1	C1	177·0 (4)	Cl1	Pt1	S2	C3	−51·0 (3)
Cl2	Pt1	S1	C2	−75·8 (3)	Cl1	Pt1	S2	C4	55·0 (3)
S2	Pt1	S1	C1	−3·1 (4)	S1	Pt1	S2	C3	129·6 (3)
S2	Pt1	S1	C2	104·1 (3)	S1	Pt1	S2	C4	−124·4 (3)

the C atoms to which they are attached. The final cycle of refinement included 83 parameters and converged to $R = 0{\cdot}034$, extinction coefficient $g = 3{\cdot}23\,(7) \times 10^{-7}$ where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Anomalous-dispersion effects were included in F_c with values taken from *International Tables for X-ray Crystallography* (1974). The highest peak in the final difference Fourier map had a height of 1·9 (2) e Å^{-3}, and the minimum negative peak had a depth of −1·6 (2) e Å^{-3}, both near the Pt atom. Final positional and equivalent isotropic displacement parameters for all non-H atoms are given in Table 2; bond lengths, angles and torsion angles are shown in Table 3.* Fig. 1 shows the molecule and the atomic numbering scheme, and Fig. 2 is a packing diagram. Programs used were *ORTEP*II (Johnson, 1976), *PLUTO*78 (Motherwell & Clegg, 1978), and *SDP*/*VAX* (Frenz, 1978).

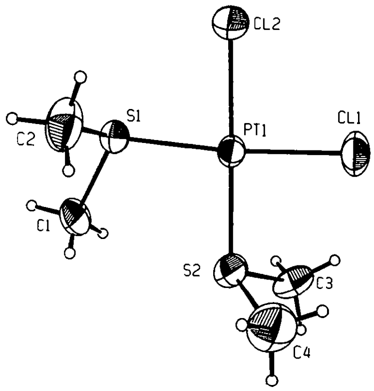

Fig. 1. The molecule of *cis*-[PtCl$_2${S(CH$_3$)$_2$}$_2$], with thermal ellipsoids drawn at the 50% probability level.

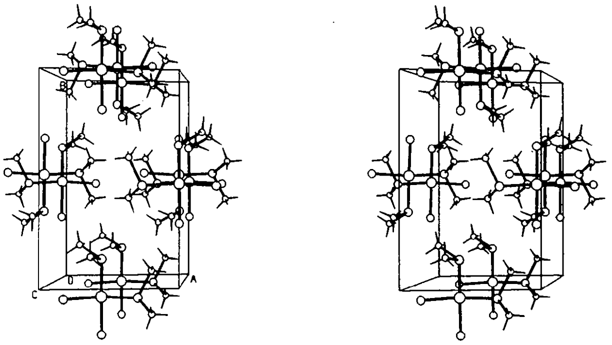

Fig. 2. Stereo packing diagram of *cis*-[PtCl$_2${S(CH$_3$)$_2$}$_2$].

Related literature. Synthesis of *cis*-dichlorobis(diethyl sulfide)platinum(II): Roulet & Barbey (1973); structure of *trans*-dichlorobis(dimethyl sulfide)-platinum(II): Cox, Saenger & Wardlaw (1934); structure of *cis*-dichlorobis(4,4′-dichlorodiphenyl sulfide)platinum(II): Spofford, Amma & Senoff (1971).

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* Tables of structure factors, anisotropic thermal parameters, H-atom positions, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52227 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (−)-(*R*)-[2-(Aminomethyl)pyrrolidine](1,1-cyclobutanedicarboxylato)-platinum(II) Monohydrate (DWA-2114R)

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Abstract. $[Pt(C_6H_6O_4)(C_5H_{12}N_2)].H_2O$, $M_r = 455{\cdot}38$, monoclinic, $P2_1$, $a = 8{\cdot}7712\,(7)$, $b = 10{\cdot}720\,(1)$, $c = 7{\cdot}3221\,(6)$ Å, $\beta = 93{\cdot}03\,(1)°$, $V = 687{\cdot}5\,(2)$ Å^3, $Z = 2$, $D_x = 2{\cdot}200$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 103{\cdot}266$ cm^{-1}, $F(000) = 436$, $T = 298$ K, final $R = 0{\cdot}020$ for 2700 unique reflections $[F_o^2 > 2\sigma(F_o^2)]$. DWA-2114R is a square-planar Pt complex with the dicarboxylate chelate ring in a boat conformation and with the aminomethylpyrrolidine chelate ring in an envelope conformation. The cyclobutane ring adopts a puckered conformation. The absolute configuration was determined by the Bijvoet method.

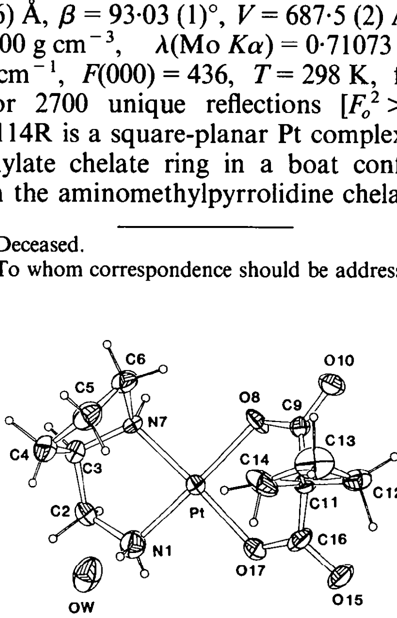

Fig. 1. A perspective view of the molecule with the numbering scheme.

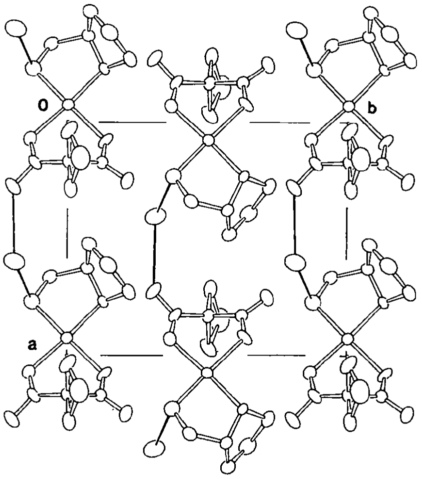

Fig. 2. Packing diagram for the title compound. Hydrogen bonds are shown as narrow lines.

Experimental. Colorless prisms of title compound were grown from an aqueous solution. $[\alpha]_D^{20°C} = -41{\cdot}7°$ (water). Crystal size $0{\cdot}20 \times 0{\cdot}13 \times 0{\cdot}10$ mm, Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, θ–2θ scan with scan speed $1{\cdot}65$–$4{\cdot}12°$ min^{-1} in θ, scan width $(0{\cdot}50 + 0{\cdot}15\tan\theta)°$. Range of indices, $-14 \le h \le 14$, $0 \le k \le 17$, $0 \le l \le 11$ ($\theta < 35°$). Lattice constants determined based on 25 2θ values ($11 < \theta < 18°$). Variation of standard $<2{\cdot}5\%$; 3340 reflections measured; 2700 observed reflections with $F_o^2 > 2\sigma(F_o^2)$. Systematic absences $0k0$, k odd. Empirical corrections for absorption (North, Phillips & Mathews, 1968); min., max. transmission coefficients 0·937, 1·000. Structure solved by the heavy-atom method. Refined by full-matrix least squares. The locations of 18 H atoms were calculated. Non-H atoms refined with anisotropic thermal parameters, but H atoms with fixed isotropic thermal parameters ($B = 5{\cdot}0$ Å^2). $\Sigma w(|F_o|$

Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-*H *atoms with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}(Å^2)
Pt	0·92524 (2)	1·000	0·58856 (2)	1·989 (2)
N(1)	0·7701 (6)	0·8759 (6)	0·4950 (8)	3·3 (1)
C(2)	0·6641 (6)	0·9400 (5)	0·3607 (8)	2·83 (9)
C(3)	0·6262 (6)	1·0699 (5)	0·4250 (8)	2·56 (9)
C(4)	0·5219 (7)	1·0766 (6)	0·587 (1)	3·5 (1)
C(5)	0·6118 (8)	1·1450 (7)	0·741 (1)	3·7 (1)
C(6)	0·7268 (7)	1·2185 (6)	0·6410 (9)	3·2 (1)
N(7)	0·7694 (5)	1·1275 (4)	0·4982 (6)	1·98 (6)
O(8)	1·0797 (4)	1·1334 (4)	0·6659 (6)	2·75 (7)
C(9)	1·1725 (5)	1·1217 (5)	0·8125 (6)	1·89 (7)
O(10)	1·2508 (5)	1·2118 (4)	0·8598 (6)	3·24 (8)
C(11)	1·1709 (4)	1·0029 (8)	0·9159 (5)	2·11 (6)
C(12)	1·2849 (6)	1·0094 (6)	1·0857 (6)	2·87 (9)
C(13)	1·153 (1)	1·0446 (8)	1·204 (1)	4·8 (2)
C(14)	1·0350 (6)	1·004 (1)	1·0514 (7)	3·73 (9)
O(15)	1·2779 (5)	0·8066 (4)	0·8300 (7)	3·74 (9)
C(16)	1·1740 (6)	0·8844 (5)	0·7924 (8)	2·80 (9)
O(17)	1·0723 (4)	0·8657 (4)	0·6677 (6)	2·89 (7)
O*W*	0·5986 (6)	0·8125 (6)	0·8261 (7)	4·7 (1)

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† Deceased.

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