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(Ethylene)platinum Dichloride–Di-*t*-butyl Sulphurdiimine

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Abstract. Monoclinic, $P2_1/c$, $a=6.212(1)$, $b=17.307(1)$, $c=15.156(4)$ Å, $\beta=98.29(4)^\circ$, $C_{10}H_{22}Cl_2N_2PtS$, $Z=4$. The title complex was synthesized by Kuyper, Vrieze & Oskam (1972) by treating ethylene-platinum-chloride with di-*t*-butyl-sulphurdiimine.

The two S–N double bonds of the sulphurdiimine ligand have lengths of 1.57 (2) and 1.53 (2) Å.

Introduction. Approximately 2500 independent reflexions were collected by means of a Nonius automatic three-circle diffractometer (Cu $K\alpha$, θ – 2θ -scan) of which 1511 were above the significance level of 2.5σ . The platinum atom was located from a Patterson synthesis. A superposition map based on all symmetry-related platinum atoms revealed the other non-hydrogen atoms. The refinement was carried out by means of a block-diagonal least-squares procedure to a final R value of 8.8%. Platinum, sulphur and chlorine were refined anisotropically and carbon and nitrogen isotropically. Atomic scattering factors, including $\Delta f'$ and $\Delta f''$ for Pt, S and Cl, were taken from *International Tables for X-ray Crystallography* (1962). No attempt was made to locate the hydrogen atoms. The final parameters are given in Table 1, bond lengths in Fig. 1, and bond angles in Table 2.

Results and discussion. The X-ray analysis was carried out in order to determine the bonding between the sulphurdiimine and the platinum atom.

Pt(1), N(1), C(1) and C(2) and the middle of O(9)–C(10) are coplanar within the limits of accuracy. The ligands are bonded to the platinum atom in an approximately square configuration (Fig. 1). The

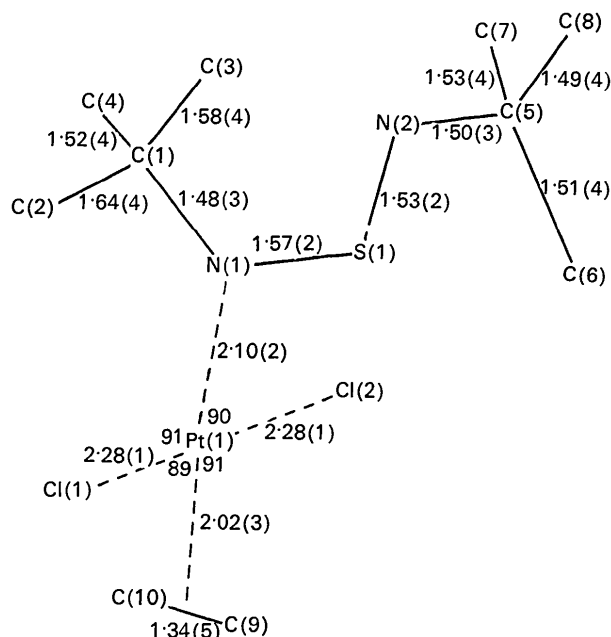


Fig. 1. Bond lengths (Å) with e.s.d.'s. The angles between the ligands of the platinum atom are indicated.

Table 1. Fractional atomic parameters ($\times 10^4$)

The thermal parameters are given as U_{ij} or U_{iso} ($\times 10^3$). E.s.d.'s are given in parentheses.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt(1)	419 (2)	3840 (1)	3924 (1)	58 (1)	52 (1)	35 (1)	11 (1)	6 (1)	10 (1)
Cl(1)	–2085 (14)	2973 (4)	4288 (5)	82 (6)	73 (4)	72 (4)	2 (4)	21 (4)	22 (4)
Cl(2)	2896 (14)	4714 (5)	3569 (5)	77 (6)	99 (6)	69 (4)	–19 (5)	2 (4)	23 (4)
S(1)	–2357 (11)	4805 (3)	2593 (3)	66 (4)	39 (3)	36 (3)	11 (3)	–1 (3)	1 (2)

Table 1 (cont.)

	x	y	z	U_{11}
N(1)	-1354 (28)	3970 (8)	2642 (10)	29 (4)
N(2)	-3448 (32)	5032 (10)	1655 (12)	44 (5)
C(1)	-1467 (42)	3431 (14)	1880 (15)	46 (6)
C(2)	17 (51)	2689 (15)	2275 (18)	63 (7)
C(3)	-120 (54)	3801 (16)	1178 (19)	75 (9)
C(4)	-3789 (52)	3196 (16)	1516 (18)	68 (8)
C(5)	-4500 (40)	5812 (12)	1532 (14)	39 (5)
C(6)	-4410 (50)	6272 (14)	2382 (18)	62 (7)
C(7)	-6868 (57)	5660 (18)	1125 (20)	77 (9)
C(8)	-3323 (62)	6172 (17)	845 (22)	81 (9)
C(9)	1316 (50)	4041 (15)	5312 (18)	61 (7)
C(10)	2615 (56)	3486 (19)	5064 (19)	77 (8)

Table 2. Bond angles with e.s.d.'s

N(1)-S(1)-N(2)	113 (1) ^o
S(1)-N(1)-C(1)	125 (1)
S(1)-N(1)-Pt(1)	107 (1)
C(1)-N(1)-Pt(1)	127 (1)
S(1)-N(2)-C(5)	118 (1)
N(1)-C(1)-C(2)	104 (2)
N(1)-C(1)-C(3)	107 (2)
N(1)-C(1)-C(4)	113 (2)
C(2)-C(1)-C(3)	104 (2)
C(2)-C(1)-C(4)	112 (2)
C(3)-C(1)-C(4)	115 (2)
N(2)-C(5)-C(6)	114 (2)
N(2)-C(5)-C(7)	106 (2)
N(2)-C(5)-C(8)	102 (2)
C(6)-C(5)-C(7)	110 (2)
C(6)-C(5)-C(8)	114 (2)
C(7)-C(5)-C(8)	109 (2)

sulphurdiimine group is N-bonded to the metal and situated opposite to the ethylene molecule. The

ethylene bond is perpendicular (86)^o to the coordination plane of the platinum atom, the sulphurdiimine molecule makes an angle of 71^o with this plane.

The sulphurdiimine group provides two independent measure values for the length of the rare double bond between sulphur and nitrogen. This bond was estimated to be 1.54 Å (Goehring, 1956) and up till now only two determinations of 1.53 and 1.54 Å (Webb & Gloss, 1967; Neidle & Rogers, 1970) were known. Our average S-N-length of 1.55 (2) is in agreement with these values. For comparison the single bond S-N has a length of 1.63 (1) Å.

The di-t-butyl-sulphurdiimine molecule is in the *cis-trans* conformation. C(6) of the t-butyl group C(5)C(6)C(7)C(8) eclipses the double bond S(1)-N(2). In the other t-butyl group C(2) eclipses the ligand bond Pt(1)-N(1).

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The unit cell of a mixed crystal of guanine and 8-azaguanine. By JOHN J. MADDEN,* *Crystallography Department, University of Pittsburgh, Pittsburgh, Pa. 15213, U.S.A.*

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Guanine (C₅H₅N₅O), 8-azaguanine (C₄H₄N₆O), and water co-crystallize in the space group P2₁/c, with $a=3.56$ (3), $b=11.37$ (11), $c=16.32$ (16) Å, $\beta=95.33$ (9)^o, and $Z=4$, a form isomorphous with the published structure of 8-azaguanine monohydrate.

A mixed crystal of guanine and 8-azaguanine was prepared by slowly cooling a warm equimolar solution of these compounds in 1M acetic acid. These crystals are isomorphous

with the published structure of 8-azaguanine (Macintyre, Singh & Werkema, 1965; Sletten, Sletten & Jensen, 1968) as judged by a comparison of the cell parameters for these compounds (Table 1) and the structure factor amplitudes of reflections on two zero-layer precession projectors (a^*b^* and a^*c^*). The intensities for these reflections were visually estimated as absent, weak, medium, and strong and struc-

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