

angles in this complex are slightly smaller than those in $[\text{Ni}(\text{DOH})_2\text{Pn}-\text{H}]^+$, but slightly larger than those in $[(\text{Cu}\{(\text{DOH})_2\text{Bn}-\text{H}\})_2]^{2+}$ (Table 3).

It is now well established that the $\text{O}\cdots\text{O}$ distance in the complex of diazadioxime varies as a function of (1) the size of the metal ion, (2) the constraint imposed by the methylene carbons bridging the amine N atoms, and (3) changing from an sp^3 amine nitrogen to an sp^2 imine nitrogen (Liss & Schlemper, 1975; Pal, Murmann, Schlemper, Fair & Hussain, 1986). As shown in Table 3, the $\text{O}\cdots\text{O}$ distance in the present complex is about 0.18 \AA shorter than that in $[\text{Cu}(\text{DOH})_2\text{Bn}-\text{H}]^+$, consistent with the smaller size of nickel(II). The $\text{O}\cdots\text{O}$ distance in the present complex is similar to that in $[\text{Ni}(\text{DOH})_2\text{Pn}-\text{H}]^+$. These results indicate the bridge in $(\text{DOH})_2\text{Bn}-\text{H}$ is essentially the same as the propylene bridge in $(\text{DOH})_2\text{Pn}-\text{H}$. Similar results were also found in the copper(II) complexes (Liss & Schlemper, 1975; Pal, Murmann, Schlemper, Fair & Hussain, 1986).

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Bis(pentafluorophenyl)(*N,N,N',N'*-tetramethylethylenediamine)platinum(II)

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Abstract. $[\text{Pt}(\text{C}_6\text{F}_5)_2(\text{tmen})]$, $\text{C}_{18}\text{H}_{16}\text{F}_{10}\text{N}_2\text{Pt}$, $M_r = 645.5$, monoclinic, $C2/c$, $a = 19.526$ (6), $b = 8.554$ (1), $c = 13.282$ (2) \AA , $\beta = 113.43$ (2) $^\circ$, $V = 2036$ (3) \AA^3 , $Z = 4$, $D_x = 2.106 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.7107 \text{ \AA}$, $\mu = 70.21 \text{ cm}^{-1}$, $F(000) = 1224$, $T = 293$ (1) K, $R = 0.040$ for 1906 observed reflections. The Pt atom in the title compound, which lies on a crystallographic twofold axis, exists in a distorted square planar geometry comprised of a chelating tetramethylethylenediamine (tmen) ligand [Pt—N 2.141 (5) \AA] and two pentafluorophenyl groups [Pt—C 2.006 (6) \AA] which make an angle of 87.1° with the square plane.

Introduction. In synthetic studies of polyfluorophenyl complexes of platinum, the title compound $[\text{Pt}(\text{C}_6\text{F}_5)_2(\text{tmen})]$ was prepared (Deacon & Nelson-

Reed, 1987). Subsequently, crystals of this compound have been obtained and the structure investigated in order to provide detailed information on the Pt coordination geometry and thereby provide additional data on the relative *trans* influence of the pentafluorophenyl ligand.

Experimental. $[\text{Pt}(\text{C}_6\text{F}_5)_2(\text{tmen})]$ was prepared as in the literature (Deacon & Nelson-Reed, 1987) and crystals were obtained by the slow evaporation of an ether/petroleum ether solution of the compound; m.p. $573\text{--}575 \text{ K}$ (dec.). Enraf-Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\alpha$ radiation; $\omega:2\theta$ scan technique. Cell parameters by least squares on 25 reflections ($11 \leq \theta \leq 20^\circ$) (de Boer & Duisenberg, 1984) on a crystal $0.14 \times 0.28 \times 0.56 \text{ mm}$. Analytical absorption correction applied; max. and min. transmission factors 0.345 and 0.067 (*SHELX76*; Sheldrick, 1976). 2738 reflections ($1.5 \leq \theta \leq 26.0^\circ$)

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measured in the range $-24 \leq h \leq 24$, $0 \leq k \leq 10$, $-16 \leq l < 3$. No significant variation in the net intensity of two reference reflections ($10\bar{2}8$, 936) measured every 7200 s. 2010 unique reflections (R_{int} 0.096) and 1906 satisfied $I \geq 2.5\sigma(I)$. Structure solved by Patterson method, full-matrix least-squares refinement on 143 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms. H atoms were located from a difference map, fixed at these sites and assigned a common isotropic thermal parameter which was refined. At convergence $R = 0.040$, $wR = 0.041$, $w = 2.31/[\sigma^2(F) + 0.0009|F|^2]$, $S = 2.17$, $(\Delta/\sigma)_{\text{max}} \leq 0.003$, $\Delta\rho_{\text{max}} = 1.75$, $\Delta\rho_{\text{min}} = -3.80 \text{ e } \text{Å}^{-3}$; extinction correction applied such that $nz = 0.00074$ (6) (*SHELX76*, Sheldrick, 1976). Refinement in the space group Cc , *i.e.* with no molecular twofold axis, resulted in chemically non-sensible bond distances and large R values. Scattering factors for neutral Pt corrected for f' and f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99 and 149) and for the remaining atoms as incorporated in *SHELX76* (Sheldrick, 1976). All calculations on SUN4/280 computer system. Atomic parameters are given in Table 1, selected interatomic parameters in Table 2* and numbering scheme used is shown in Fig. 1 which was drawn with *ORTEP* (Johnson, 1976) at 15% probability levels.

Discussion. The Pt atom in [Pt(C₆F₅)₂(tmen)] lies on a crystallographic twofold axis such that only half the molecule comprises the asymmetric unit. The structure is molecular with the closest intermolecular contact occurring between the F(6) atom and a

* Lists of structure factors, anisotropic thermal parameters, mean-plane data, all interatomic parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54048 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

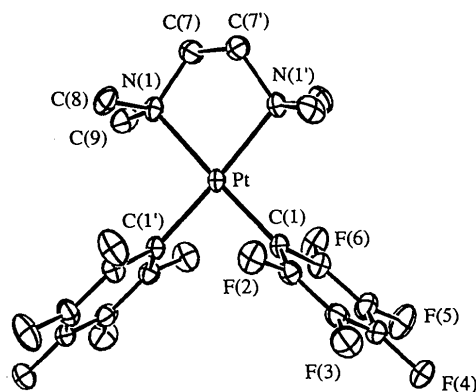


Fig. 1. Molecular structure and crystallographic numbering scheme employed for [Pt(C₆F₅)₂(tmen)] (Johnson, 1976).

Table 1. Fractional atomic coordinates and B_{eq} values (Å^2)

	$B_{\text{eq}} = (8\pi^2/3) \text{ trace } U.$			B_{eq}
	x	y	z	
Pt	0.0	-0.2020 (4)	0.25	2.47
F(2)	-0.1120 (3)	-0.2119 (6)	0.0423 (3)	5.30
F(3)	-0.2190 (3)	-0.4185 (7)	0.0220 (5)	5.77
F(4)	-0.2439 (4)	-0.5105 (6)	0.2000 (8)	7.11
F(5)	-0.1598 (3)	-0.3890 (9)	0.4001 (5)	8.26
F(6)	-0.0525 (3)	-0.1838 (7)	0.4227 (3)	5.60
N(1)	0.0779 (3)	0.1658 (7)	0.2708 (4)	3.13
C(1)	-0.0765 (3)	-0.1859 (7)	0.2336 (5)	2.86
C(2)	-0.1216 (3)	-0.2517 (8)	0.1339 (5)	3.42
C(3)	-0.1769 (3)	-0.3598 (9)	0.1217 (6)	4.10
C(4)	-0.1903 (4)	-0.4054 (8)	0.2114 (8)	4.74
C(5)	-0.1471 (4)	-0.3444 (9)	0.3124 (6)	4.71
C(6)	-0.0917 (4)	-0.2372 (9)	0.3209 (6)	3.68
C(7)	0.0403 (4)	0.3103 (8)	0.2884 (6)	4.26
C(8)	0.0958 (4)	0.1822 (10)	0.1738 (7)	4.71
C(9)	0.1489 (4)	0.1459 (10)	0.3700 (6)	4.22

Table 2. Selected interatomic distances (Å) and bond angles ($^\circ$)

Pt—N(1)	2.141 (5)	Pt—C(1)	2.006 (6)
N(1)—C(7)	1.502 (9)	N(1)—C(8)	1.470 (9)
N(1)—C(9)	1.495 (8)	C(7)—C(7)*	1.50 (1)
C(1)—C(2)	1.385 (8)	C(1)—C(6)	1.379 (9)
C(2)—C(3)	1.383 (9)	C(3)—C(4)	1.37 (1)
C(4)—C(5)	1.37 (1)	C(5)—C(6)	1.39 (1)
C(2)—F(2)	1.347 (7)	C(3)—F(3)	1.347 (8)
C(4)—F(4)	1.341 (9)	C(5)—F(5)	1.339 (8)
C(6)—F(6)	1.342 (8)		
N(1)—Pt—N(1')	84.0 (3)	N(1)—Pt—C(1)	176.6 (2)
N(1)—Pt—C(1')	92.9 (2)	C(1)—Pt—C(1')	90.1 (3)
Pt—N(1)—C(7)	105.4 (4)	Pt—N(1)—C(8)	111.2 (4)
Pt—N(1)—C(9)	113.1 (4)	C(7)—N(1)—C(8)	111.0 (6)
C(7)—N(1)—C(9)	107.2 (6)	C(8)—N(1)—C(9)	108.8 (6)
Pt—C(1)—C(2)	123.6 (5)	Pt—C(1)—C(6)	122.3 (5)
N(1)—C(7)—C(7')	110.1 (6)		

* Primed atoms are related by twofold axis.

symmetry related ($x, \bar{y}, \frac{1}{2} + z$) methyl bound H atom such that F(6)···H(83) 2.40 Å , a value which is less than the sum of the van der Waals radii for these two atoms of 2.70 Å (Bondi, 1964) but too long to be considered a significant hydrogen-bonding interaction.

The Pt atom in the complex is coordinated by a chelating tmen ligand and two pentafluorophenyl groups. The geometry about the metal atom is square planar with the N(1), N(1') and C(1), C(1') donor atoms lying ± 0.023 (5) and ± 0.030 (6) Å , respectively, out of the PtN₂C₂ least-squares plane. The pentafluorophenyl groups make an angle of 87.1° with the square plane in order to minimize steric interactions between the C₆F₅ groups: the dihedral angle between the two pentafluorophenyl groups is 96.5°. The conformation of the NCCN backbone of the tmen ligand is δ .

There are several structures in the literature containing the Pt(tmen) moiety available for comparison with the title compound. In the symmetrical dications, [PtL₂(tmen)] ($L = 9$ -methylguanine,

9-ethylguanine, 1,3-dimethylxanthine and 1,3,9-trimethylxanthine), the Pt—N bond distances involving the tmen ligand lie in the range 2.00 (4) to 2.075 (6) Å (Orbell, Taylor, Birch, Lawton, Vilkins & Keefe, 1988). In the perchlorate salt of the [PtCl(H₂C=CH₂)(tmen)] cation, the Pt—N bond distance *trans* to the Cl atom is 2.084 (2) Å and the other Pt—N distance is 2.119 (2) Å (Gervasio, Mason, Maresca & Natile, 1986). Similarly in the structure of [PtCl(1-methylcytosine)(tmen)]ClO₄ (Preut, Frommer & Lippert, 1990), the Pt—N bond *trans* to the Cl atom is 2.078 (4) Å and the other Pt—N distance, *trans* to the nucleobase, is 2.066 (4) Å. The Pt—N bond distance of 2.141 (5) Å found in [Pt(C₆F₅)₂(tmen)] is significantly longer than in any of complexes cited above and reflects the greater *trans* influence of the pentafluorophenyl groups (Appleton, Clark & Manzer, 1973).

There are two other compounds containing the *cis*-Pt(C₆F₅)₂ entity that have been structurally characterized. In the structure of [Pt(C₆F₅)₂(C₆H₅C≡CC₆H₅)₂], a molecule with a two-fold symmetry axis, the Pt—C bond distance involving the pentafluorophenyl group is 2.048 (3) Å (Usón, Forniés, Tomás, Menjón & Welch, 1986). In the structure of [Pt(C₆F₅)₂{SC(S)P(C₆H₁₁)₃}(CO)], the Pt—C bond distance *trans* to the S atom is 2.056 (6) Å and the other Pt—C distance is 2.045 (7) Å (Usón, Forniés, Usón, Yagüe, Jones & Meyer-Bäse, 1986). The relatively long Pt—C bond distances in these structures may be contrasted to the Pt—C bond distance of 2.006 (6) Å found in [Pt(C₆F₅)₂(tmen)]. This study demonstrates that the

tmen ligand has a weaker *trans* influence than an acetylene molecule.

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Structure of Potassium Thiosaccharinate Monohydrate*

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Abstract. K[C₇H₄NO₂S₂].H₂O, *M*_r = 255.36, orthorhombic, *Pbca*, *a* = 8.873 (1), *b* = 27.108 (6), *c* = 8.708 (1) Å, *V* = 2094.5 Å³, *Z* = 8, *D*_x =

1.62 Mg m⁻³, λ(Mo Kα) = 0.71069 Å, μ = 0.81 mm⁻¹, *F*(000) = 1032, room temperature, final *R* = 0.032 for 1801 observed unique reflections. The structure is built up of K⁺ cations, thiosaccharinate anions and water molecules. The K⁺ ion is six

* Thiosaccharin is 1,2-benzisothiazol-3(2*H*)-thione 1,1-dioxide.