

Fig. 1. The molecular structure of the title compound.

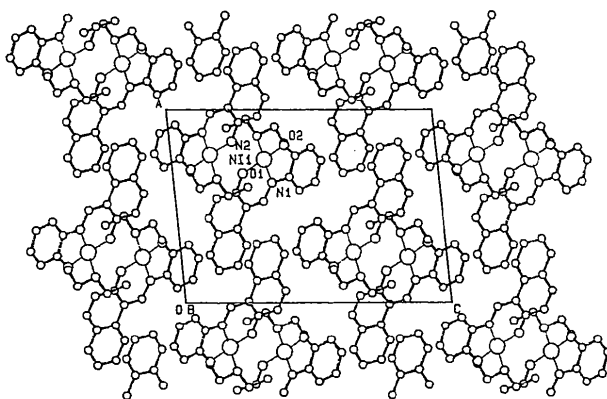


Fig. 2. Packing of the molecules.

Table 3. Bond lengths (Å) in some nickel(II) complexes

	(I)	(II)	(III)	(IV)
C(1)—C(2)	1.402 (6)	1.401 (7)	1.422 (8)	1.401 (5)
C(1)—C(11)	1.430 (5)	1.429 (4)	1.446 (6)	1.416 (8)
C(2)—O(2)	1.323 (5)	1.318 (5)	1.332 (5)	1.296 (5)
C(11)—N(1)	1.298 (5)	1.302 (5)	1.292 (5)	1.303 (5)
C(12)—C(13)			1.410 (6)	1.393 (5)
C(12)—N(1)			1.434 (7)	1.431 (4)
C(13)—O(2)			1.334 (6)	1.339 (4)
N(1)—Ni(1)	1.913 (4)	1.918 (4)	1.941 (3)	1.848 (4)
N(2)—Ni(1)			1.947 (4)	1.955 (4)
O(1)—Ni(1)	1.829 (2)	1.828 (2)	1.828 (3)	1.804 (4)
O(2)—Ni(1)			1.830 (4)	1.842 (3)

(I) Bis[*N*-(1,3-dioxolan-2-yl)methyl]salicylidiminato]nickel(II) (Fernandez-G., Rosales-Hoz, Rubio-Arroyo, Salcedo, Toscano & Vela, 1987); (II) bis[*N*-(1,3-dioxolan-2-yl)-3-hydroxy-1-naphthalidiminato]nickel(II) (Fernandez-G. *et al.*, 1987); (III) {1-[(2-hydroxyphenyl)iminomethyl]-naphthalen-2-olato-*O,O',N'*}piperidinenickel(II) (Elerman *et al.*, 1991); (IV) present work.

planar coordination. The coordination of the ligands around the Ni atom is fairly planar, the bonding angles of the ligands are between 86.8 and 94.8°. The Ni atom is 0.015 Å out of the plane of its ligands. The angles between the planar organic groups are smaller than 9°. The Ni atom deviates from these planes by less than 0.10 Å.

The diethylamino ligand is fully extended perpendicular to the Ni coordination plane.

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A *gem*-Diolato Complex of Platinum: Structure of {P(C₆H₅)₃}₂Pt{O₂C(CF₃)₂}

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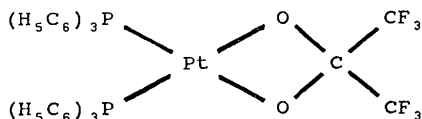
Abstract. Hexafluoropropane-2,2-diolatobis(tri-phenylphosphine)platinum(II), C₃₉H₃₀F₆O₂P₂Pt, *M*,

= 901.70, monoclinic, *P*2₁/*n*, *a* = 14.027 (2), *b* = 21.127 (3), *c* = 12.229 (2) Å, β = 100.56 (1)°, *V* = 3563 (2) Å³, *Z* = 4, *D*_m = 1.71 (3), *D*_x = 1.681 Mg m⁻³, *F*(000) = 1768, λ(Mo *K*α) =

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0.71073 Å, $\mu = 4.13 \text{ mm}^{-1}$, $T = 295 \text{ K}$. 4112 unique reflections with $I > 2.5\sigma(I)$ were used to refine 272 variables on F giving agreement factors of $R = 0.034$ and $wR = 0.046$. The alkoxide ligand coordinates to platinum through *gem*-diolate atoms to form a planar four-membered chelate ring, with a bite angle of $66.7(2)^\circ$ and Pt—O bond lengths of 2.040 (5) and 2.056 (5) Å.

Introduction. The *gem*-diol $(\text{CF}_3)_2\text{C}(\text{OH})_2$ has a rich coordination chemistry. It can act as a chelating ligand in two modes: as the dialkoxide $[\text{O}-\text{C}(\text{CF}_3)_2-\text{O}]^{2-}$, (OCO), forming a four-membered ring (Bradford, Hynes, Payne & Willis, 1990), and as the condensed dialkoxide $[\text{O}-\text{C}(\text{CF}_3)_2-\text{O}-\text{C}(\text{CF}_3)_2-\text{O}]^{2-}$, (OCOCO), forming a six-membered ring (Modinos & Woodward, 1975). A third coordination geometry has recently been described (Hynes, Payne & Willis, 1990) in which the ligand behaves as a monodentate alkoxide. Finally, Mitchell & Stone (1972) formulated the ligand as a dialkoxide bridging two Au atoms. We have developed a rational synthetic approach to complexes of this ligand (Bradford *et al.*, 1990), and describe here the structure of $\{\text{P}(\text{C}_6\text{H}_5)_3\}_2\text{Pt}\{\text{O}_2\text{C}(\text{CF}_3)_2\}$, a Pt complex containing the ligand as a chelating dialkoxide, which was first prepared by Hayward & Nyman (1971).



Experimental. A sample of the complex was prepared by the method of Bradford *et al.* (1990), and recrystallized from chloroform by layering with hexane. Pale yellow-green rectangular needles, crystal density by neutral buoyancy in 1,2-dibromoethane and pentane. Weissenberg and precession photography showed Laue symmetry $2/m$, systematic absences $h0l$, $h + l$ odd, and $0k0$, k odd, defined space group $P2_1/n$, No. 14. Crystal used for recording intensity data of approximate dimensions $0.40 \times 0.35 \times 0.30 \text{ mm}$, $V = 0.0102 \text{ mm}^3$, 12 crystal faces $\{010\}$, $\{001\}$, $\{11\bar{1}\}$, $(1\bar{1}1)$, $(1\bar{1}0)$, $(\bar{1}\bar{1}0)$ and (110) . Enraf-Nonius (1984) CAD-4F diffractometer, cell parameters from 20 reflections with $14 < \theta < 17^\circ$, Mo radiation monochromatized by graphite. ω scans had an average width at half-height of 0.12° . 6927 observations ($h, k, \pm l$) by $\theta-2\theta$ scan method, variable speed, dispersion corrected scans of width 0.7° , 25% extensions at both ends for backgrounds. Maximum time per datum 60 s, maximum 2θ 50° , over a period of 103 h. Index ranges 0 to 16, -1 to 25, and -14 to 14. Standard reflections 411, 400, $\bar{1}05$ and 002, monitored every 3 h of X-ray exposure time, 160 measurements, deviations random, no decay correction

applied. Background, monochromator polarization, crystal polarization and Lorentz corrections applied, Enraf-Nonius (1983) *Structure Determination Package* running on a DEC PDP 11/23+ computer. Analytical absorption correction (Cahen & Ibers, 1972), transmission factors from 0.468 to 0.589. R factor for averaging 821 observations on F 0.017. 5735 unique reflections, 5159 with $I > 0$ for solution by *MULTAN*11/82 (Main *et al.*, 1982) and Fourier techniques. Parameters refined by full-matrix least-squares methods, minimizing $\sum w(|F_o| - |F_c|)^2$, weight $w = 4F_o^2/\sigma^2(F_o^2)$. With all 50 non-H atoms included, refinement converged at agreement factors $R = 0.047$ and $wR = 0.063$. Of the 30 phenyl H atoms, 27 were located among the first 35 peaks of a difference Fourier synthesis, peak heights from 0.52 (14) to 0.23 (14) $\text{e} \text{ \AA}^{-3}$. All included with idealized sp^2 geometries, C—H 0.95 Å, isotropic Debye parameters 110% of those of bonded atoms, positions updated as refinement progressed but not refined. Refinement completed using *SHELX76* (Sheldrick, 1976), Pt scattering factor from *International Tables for X-ray Crystallography* (1974, Vol. IV), real component of anomalous scattering included for all non-H atoms (Cromer & Liberman, 1970). Phenyl ring C atoms refined with isotropic Debye factors, H atoms riding in idealized positions, all other atoms assigned anisotropic thermal parameters. Evidence for secondary extinction, parameter refined to $6.1(3) \times 10^{-4}$, weight $k = 0.813$, $g = 8.75 \times 10^{-4}$. Convergence with updated H-atom positions at $R = 0.034$ and $wR = 0.046$, 4112 unique observations with $I > 2.5\sigma(I)$, 272 variables, $(\Delta/\sigma)_{\text{max}} 0.01$. Residual electron density maximum $0.81 \text{ e} \text{ \AA}^{-3}$ (0.262, 0.383, 0.148) between Pt and P(2), no chemical significance. Analysis of variance showed no unusual trends. Final atomic positional parameters are given in Table 1.*

Discussion. The crystal structure consists of discrete molecules. The shortest intermolecular distances of approach are: H...H, 2.44 Å between H13 and H66 at $(\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z)$ and H...F, 2.50 Å between H23 and F(5) at $(\frac{1}{2} + x, \frac{1}{2} - y, z - \frac{1}{2})$. A perspective view showing the atom-numbering scheme is presented in Fig. 1. Selected bond distances and bond angles are given in Table 2.

A distorted square-planar coordination geometry at the Pt atom is formed by the phosphine ligands

* Lists of H-atom parameters, anisotropic thermal parameters, root-mean-square amplitudes of vibration, supplementary dimensions, weighted least-squares planes, selected torsion angles and structure amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54555 (38 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ($\times 10^4$) and thermal parameters ($\times 10^3$) with *e.s.d.*'s in parentheses

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

	x	y	z	$U_{eq} (\text{\AA}^2)$
Pt	2312.3 (2)	3360.6 (1)	2384.7 (2)	33.68 (9)
P(1)	2104.3 (13)	2322.7 (8)	2010.2 (15)	33.6 (5)
P(2)	3706.7 (13)	3495.9 (8)	1757.4 (14)	33.0 (5)
F(1)	550 (4)	5165 (3)	2521 (6)	108 (3)
F(2)	-414 (4)	4399 (3)	2674 (5)	94 (2)
F(3)	395 (5)	4399 (4)	1354 (6)	115 (3)
F(4)	2087 (4)	4021 (3)	4990 (5)	85 (2)
F(5)	640 (4)	4362 (3)	4800 (5)	86 (2)
F(6)	1773 (4)	4971 (2)	4506 (5)	93 (2)
O(1)	1092 (4)	3531 (2)	3049 (5)	52 (2)
O(2)	2135 (3)	4292 (2)	2740 (5)	51 (2)
C(1)	1284 (5)	4169 (3)	3126 (7)	45 (2)
C(2)	444 (6)	4532 (5)	2420 (9)	68 (3)
C(3)	1432 (6)	4387 (4)	4346 (8)	58 (3)
C(11)	2527 (5)	1795 (3)	3169 (6)	39 (2)
C(12)	2428 (6)	1144 (4)	3081 (7)	60 (2)
C(13)	2707 (7)	761 (5)	3996 (8)	76 (3)
C(14)	3086 (8)	1018 (5)	5012 (10)	91 (3)
C(15)	3188 (8)	1674 (5)	5119 (9)	82 (3)
C(16)	2912 (6)	2056 (4)	4199 (7)	56 (2)
C(21)	2552 (5)	2033 (3)	793 (5)	35 (2)
C(22)	3183 (5)	1537 (3)	790 (6)	44 (2)
C(23)	3514 (6)	1376 (4)	-176 (6)	53 (2)
C(24)	3188 (6)	1703 (4)	-1142 (7)	58 (2)
C(25)	2552 (6)	2189 (4)	-1159 (7)	52 (2)
C(26)	2237 (5)	2366 (3)	-196 (5)	35 (2)
C(31)	815 (5)	2118 (3)	1726 (5)	37 (2)
C(32)	296 (5)	2209 (3)	2589 (6)	47 (2)
C(33)	-657 (6)	2025 (4)	2452 (7)	55 (2)
C(34)	-1121 (6)	1752 (3)	1464 (6)	49 (2)
C(35)	-618 (5)	1670 (3)	609 (6)	45 (2)
C(36)	352 (5)	1852 (3)	735 (6)	40 (2)
C(41)	4497 (5)	2821 (3)	1681 (5)	35 (2)
C(42)	4712 (5)	2438 (3)	2621 (6)	41 (2)
C(43)	5332 (6)	1928 (4)	2653 (7)	51 (2)
C(44)	5762 (6)	1793 (4)	1748 (7)	57 (2)
C(45)	5569 (6)	2174 (4)	823 (7)	58 (2)
C(46)	4934 (6)	2682 (4)	778 (7)	50 (2)
C(51)	3465 (5)	3901 (3)	424 (6)	40 (2)
C(52)	4248 (6)	4157 (4)	-34 (7)	54 (2)
C(53)	4021 (7)	4493 (4)	-1036 (8)	71 (3)
C(54)	3103 (7)	4578 (5)	-1535 (8)	76 (3)
C(55)	2342 (7)	4346 (4)	-1102 (8)	69 (2)
C(56)	2537 (6)	4006 (4)	-117 (6)	50 (2)
C(61)	4511 (5)	4053 (3)	2620 (6)	35 (2)
C(62)	5473 (5)	3909 (4)	3048 (6)	45 (2)
C(63)	6068 (6)	4357 (4)	3685 (6)	54 (2)
C(64)	5706 (6)	4942 (4)	3881 (6)	52 (2)
C(65)	4767 (6)	5100 (4)	3450 (6)	55 (2)
C(66)	4165 (6)	4654 (4)	2820 (6)	47 (2)

oxide bite angle of $86.5(4)^\circ$ for the condensed dialkoxide ligand which forms a six-membered ring in $\{P(C_6H_5)_3\}_2Pt\{OCOCO\}$. The P—Pt—P angle of $98.25(6)^\circ$ is slightly smaller than that seen in the carbonato analog $\{P(C_6H_5)_3\}_2Pt\{O_2CO\}$, $99.25(5)^\circ$, by Gregg *et al.* (1988).

The phenyl rings in the triphenylphosphine ligands adopt a conformation very similar to that noted by Gregg *et al.* Thus H16 (at 2.95 Å) and H56 (at 2.83 Å) might be considered to fill the fifth and sixth coordination sites of a highly distorted octahedron at Pt. However, unlike the carbonato analog, these approaches do not affect the Pt—P bond lengths. The mean P—C distance is 1.822(6) Å, the Pt—P—C angles range from $109.6(2)$ to $119.7(2)^\circ$ and the C—P—C angles from $100.0(3)$ to $109.8(3)^\circ$, indicative of steric crowding in the solid state. C—C distances, ranging from 1.332(12) to 1.427(10) Å, and C—C—C angles, ranging from $117.8(8)$ to $122.2(10)^\circ$, have been deposited.

The four-membered chelate ring is planar within experimental error (see deposition footnote). Dimensions within the OCO ligand are very similar to those we found in the Ni and Cu complexes (Bradford *et al.*, 1990). The mean O—C bond length is 1.380(6) and the mean C—C value 1.536(4) Å, with a mean Pt—O—C angle of $92.0(3)^\circ$. The CF₃ groups behaved well during the refinement. The mean C—F distance is 1.333(10) Å, the mean F—C—F angle $106.2(8)^\circ$, whilst C—C—F angles range from $110.2(8)$ to $114.7(7)^\circ$, values no different from those found in our earlier studies. The major difference between the OCO and OCOCO complexes is that the alkoxide C—O bond lengths are somewhat longer in the OCO species.

and the O-bound dialkoxide, with the Pt, P, P plane twisted by $5(1)^\circ$ from that of Pt, O, O. The bite angle of the dialkoxide at the metal, $66.7(2)^\circ$, is very similar to that of $67.1(1)^\circ$ which we found in the complex (mac)Ni(OCO), where mac is a macrocyclic triamine (Bradford *et al.*, 1990). A similar Pt—O—C—O four-membered ring is present in carbonato complexes of Pt^{II}, $(R_3P)_2Pt(CO_3)$, and slightly smaller O—Pt—O bite angles have been reported: 64 and $64.5(2)^\circ$ (R = phenyl; Cariati, Mason, Robertson & Ugo, 1967; Gregg, Powell & Sawyer, 1988; respectively), $64.2(1)^\circ$ (R = isopropyl; Robertson & Tucker, 1983), and $63.8(2)^\circ$ (R = ethyl; Davies, Eagle, Pinkerton & Syed, 1987). The mean Pt—P distance is 2.247(1) Å, and the Pt—O distances 2.040(5) and 2.056(5) Å. Modinos & Woodward (1975) found Pt—P distances of 2.03(3) and 2.04(1) Å, a P—Pt—P angle of $98.5(1)^\circ$ and a dialk-

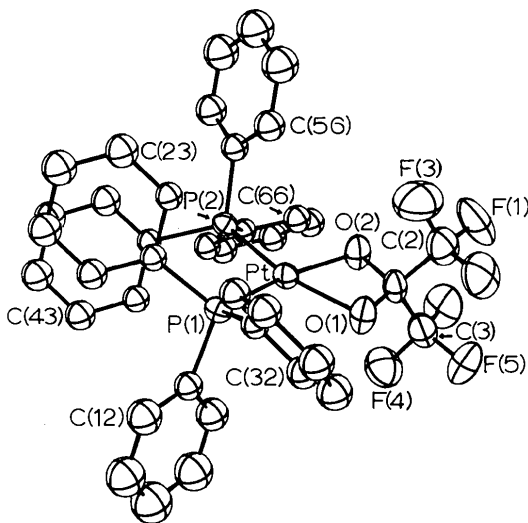


Fig. 1. A perspective view of the molecule, showing the atom-numbering scheme.

Table 2. Intramolecular bond distances (Å) and angles (°)

Pt—O(1)	2.056 (5)	Pt—O(2)	2.040 (5)
Pt—P(1)	2.248 (2)	Pt—P(2)	2.246 (2)
O(1)—C(1)	1.375 (8)	O(2)—C(1)	1.385 (8)
C(1)—C(2)	1.533 (12)	C(1)—C(3)	1.539 (12)
C(2)—F(1)	1.349 (10)	C(3)—F(4)	1.341 (10)
C(2)—F(2)	1.328 (10)	C(3)—F(5)	1.331 (9)
C(2)—F(3)	1.322 (11)	C(3)—F(6)	1.324 (9)
P(1)—C(11)	1.815 (7)	P(2)—C(41)	1.819 (7)
P(1)—C(21)	1.825 (7)	P(2)—C(51)	1.818 (7)
P(1)—C(31)	1.830 (7)	P(2)—C(61)	1.826 (7)
P(2)—Pt—P(1)	98.24 (6)	O(1)—Pt—O(2)	66.7 (2)
O(1)—Pt—P(2)	162.26 (14)	O(2)—Pt—P(2)	95.61 (14)
O(1)—Pt—P(1)	99.49 (13)	O(2)—Pt—P(1)	165.41 (14)
Pt—O(1)—C(1)	91.8 (4)	Pt—O(2)—C(1)	92.2 (4)
O(1)—C(1)—O(2)	109.3 (5)	C(2)—C(1)—C(3)	110.5 (7)
O(1)—C(1)—C(2)	109.3 (7)	O(2)—C(1)—C(2)	109.6 (6)
O(1)—C(1)—C(3)	110.5 (6)	O(2)—C(1)—C(3)	107.7 (6)
C(1)—C(2)—F(1)	112.6 (8)	F(1)—C(2)—F(2)	106.1 (8)
C(1)—C(2)—F(2)	113.5 (8)	F(1)—C(2)—F(3)	106.6 (9)
C(1)—C(2)—F(3)	110.2 (8)	F(2)—C(2)—F(3)	107.5 (8)
C(1)—C(3)—F(4)	110.7 (7)	F(4)—C(3)—F(5)	105.5 (7)
C(1)—C(3)—F(5)	114.7 (7)	F(4)—C(3)—F(6)	105.1 (7)
C(1)—C(3)—F(6)	113.8 (7)	F(5)—C(3)—F(6)	106.3 (7)
Pt—P(1)—C(11)	115.4 (2)	C(11)—P(1)—C(21)	108.6 (3)
Pt—P(1)—C(21)	116.3 (2)	C(11)—P(1)—C(31)	100.0 (3)
Pt—P(1)—C(31)	110.8 (2)	C(21)—P(1)—C(31)	104.0 (3)
Pt—P(2)—C(41)	119.7 (2)	C(41)—P(2)—C(51)	109.8 (3)
Pt—P(2)—C(51)	109.6 (2)	C(41)—P(2)—C(61)	102.5 (3)
Pt—P(2)—C(61)	111.7 (2)	C(51)—P(2)—C(61)	102.0 (3)

This paper extends the very limited range of reported structures of complexes in which a chelating dinegative ligand is bonded to platinum through oxygen, and demonstrates again the use of highly fluorinated diols in filling two coordination sites on a metal atom.

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A Chiral Aminophosphine Complex of Platinum: Structure of *cis*-Dichloro[(1*S*)-*N,N*-dimethyl-1-{2-[(*S*)-*tert*-butylphenylphosphino]phenyl}ethylamine-*N,P*]platinum(II)

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Abstract. [Pt(C₂₀H₂₈NP)Cl₂], *M_r* = 579.42, orthorhombic, *P*2₁2₁2₁, *a* = 13.482 (1), *b* = 15.926 (4), *c* = 9.827 (2) Å, *V* = 2110 (1) Å³, *Z* = 4, *D_m* = 1.830 (3), *D_x* = 1.823 Mg m⁻³, *F*(000) = 1128, λ(Mo *K*α) = 0.71073 Å, μ = 7.05 mm⁻¹, *T* = 295 K, *R* = 0.025, *wR* = 0.029 for 3719 unique reflections with *I* > 3σ(*I*) and 227 variables refined on *F*. The four-coordinate

Pt atom displays a distorted square-planar geometry with Pt—P and Pt—N distances of 2.236 (1) and 2.106 (5) Å respectively. The Pt—Cl(1) distance of 2.360 (2) Å is longer than Pt—Cl(2) at 2.297 (2) Å due to the greater *trans* influence of P relative to N. The six-membered chelate ring adopts a λ twist-boat conformation, with *S* absolute configuration both at backbone carbon C(7) and at the coordinated P atom.

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