

Fig. 1. ORTEP (Johnson, 1976) drawing and numbering scheme. The ellipsoids are drawn at the 30% probability surface.

Related literature. The syntheses, reactions and structural aspects of allyl complexes of rhodium have been the subject of numerous reviews; recent surveys include those by Collman, Hegedus, Norton & Finke (1987) and by Hughes (1982). The structure reported here can be compared to that of a cyclooctenyl-rhodium complex (Pickardt & Stuhler, 1980) and

that of a methylallyl-rhodium complex (Hewitt & deBoer, 1968).

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(η^2 -1,3-Di-*tert*-butyl-2,5-dioxo-3-cyclopentene-1-carbonitrile)bis(triphenylphosphine)-platinum(0)–Diethyl Ether

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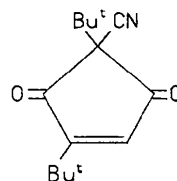
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Abstract. [Pt(C₁₄H₁₉NO₂){P(C₆H₅)₃}₂].C₄H₁₀O, $M_r = 1027.1$, triclinic, $P\bar{1}$, $a = 11.240$ (4), $b = 13.748$ (5), $c = 16.224$ (6) Å, $\alpha = 76.77$ (3), $\beta = 79.56$ (3), $\gamma = 83.09$ (3)°, $V = 2392$ (12) Å³, $Z = 2$, $D_x = 1.426$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 31.9$ cm⁻¹, $F(000) = 1044$, $T = 296$ K, $R_F = 4.11\%$ for 5014 observed reflections and 479 parameters. The structure is consistent with a Pt⁰ olefin diphosphine complex. The Pt–olefin plane is twisted 11.4° out of the PtP₂ plane by the presence of the bulky *tert*-Bu group on the cyclopentenedione ring. The dihedral angle between the ring plane and the Pt–olefin plane is 95.8°. The two Pt–C distances differ

significantly: the distance to the ring position bearing the *tert*-Bu group is 2.170 (7) Å, while the distance to the unsubstituted C atom is much shorter, 2.121 (8) Å.

Experimental. The title compound was obtained by the addition of the ligand (see below) to [Pt(C₂H₄)(PPh₃)₂] (Macklin, 1988). Yellow crystals



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Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Pt	2646.3 (3)	7807.0 (2)	3151.8 (2)	53.5 (1)
P(1)	4618 (2)	7844 (2)	3371 (1)	57 (1)
P(2)	2767 (2)	6992 (2)	2039 (1)	58 (1)
O(1)	-407 (5)	8740 (4)	2559 (4)	73 (2)
O(2)	2301 (6)	10247 (5)	3730 (4)	85 (3)
O(3)	7793 (10)	3787 (10)	1320 (9)	182 (7)
N(1)	2162 (8)	10354 (7)	1339 (6)	94 (4)
C(1)	6686 (4)	8696 (4)	2257 (4)	70 (4)
C(2)	7233	9471	1648	81 (4)
C(3)	6554	10370	1381	82 (4)
C(4)	5328	10494	1724	82 (4)
C(5)	4781	9718	2333	73 (4)
C(6)	5460	8819	2600	61 (3)
C(7)	5602 (5)	8713 (4)	4489 (4)	79 (4)
C(8)	5694	8873	5293	88 (5)
C(9)	4909	8437	6017	100 (5)
C(10)	4032	7843	5937	90 (5)
C(11)	3940	7683	5132	78 (4)
C(12)	4725	8119	4409	63 (3)
C(13)	6104 (5)	6388 (4)	2613 (3)	76 (4)
C(14)	6830	5489	2618	90 (5)
C(15)	7083	4886	3393	102 (5)
C(16)	6611	5181	4163	106 (5)
C(17)	5886	6079	4158	90 (4)
C(18)	5632	6683	3383	64 (3)
C(19)	4508 (5)	8172 (4)	947 (4)	69 (4)
C(20)	5556	8360	347	78 (4)
C(21)	6219	7582	1	93 (5)
C(22)	5834	6615	256	91 (5)
C(23)	4785	6427	856	77 (4)
C(24)	4122	7206	1201	61 (3)
C(25)	3153 (5)	5186 (4)	3166 (3)	71 (4)
C(26)	3216	4147	3454	94 (5)
C(27)	2922	3541	2951	92 (5)
C(28)	2565	3974	2159	93 (5)
C(29)	2502	5013	1870	78 (4)
C(30)	2796	5619	2374	65 (3)
C(31)	485 (5)	6833 (4)	1558 (3)	77 (4)
C(32)	-461	7175	1087	85 (4)
C(33)	-384	8055	456	86 (4)
C(34)	639	8592	295	77 (4)
C(35)	1586	8250	766	69 (4)
C(36)	1509	7371	1397	64 (3)
C(41)	772 (6)	8128 (6)	3703 (5)	58 (3)
C(42)	1599 (7)	8602 (6)	4050 (5)	62 (3)
C(43)	1707 (7)	9628 (7)	3586 (5)	66 (3)
C(44)	886 (7)	9881 (6)	2875 (5)	60 (3)
C(45)	326 (7)	8856 (6)	2996 (5)	63 (3)
C(46)	19 (7)	7279 (7)	4260 (6)	72 (4)
C(47)	694 (10)	6651 (8)	4966 (7)	100 (5)
C(48)	-248 (9)	6561 (7)	3719 (7)	92 (4)
C(49)	-1183 (9)	7745 (8)	4656 (7)	103 (5)
C(50)	-117 (8)	10776 (7)	2998 (6)	75 (4)
C(51)	464 (11)	11754 (7)	2920 (8)	111 (6)
C(52)	-945 (10)	10979 (8)	2317 (7)	103 (5)
C(53)	-895 (10)	10494 (9)	3897 (7)	108 (5)
C(54)	1598 (8)	10152 (7)	2018 (6)	70 (4)
C(55)	9757 (17)	4142 (15)	1168 (13)	224 (13)
C(56)	8776 (16)	3944 (18)	799 (14)	218 (14)
C(57)	6865 (15)	3465 (16)	1001 (11)	188 (11)
C(58)	5873 (15)	3291 (15)	1595 (11)	192 (11)

Table 2. Bond lengths (\AA)

Pt—P(1)	2.316 (2)	C(41)—C(45)	1.460 (11)
Pt—P(2)	2.310 (2)	C(41)—C(46)	1.540 (11)
Pt—C(41)	2.170 (7)	C(42)—C(43)	1.446 (11)
Pt—C(42)	2.121 (8)	C(43)—C(44)	1.558 (12)
P(1)—C(6)	1.835 (5)	C(44)—C(45)	1.570 (12)
P(1)—C(12)	1.834 (7)	C(44)—C(50)	1.588 (12)
P(1)—C(18)	1.846 (6)	C(44)—C(54)	1.465 (11)
P(2)—C(24)	1.852 (5)	C(46)—C(47)	1.530 (14)
P(2)—C(30)	1.839 (6)	C(46)—C(48)	1.548 (16)
P(2)—C(36)	1.861 (6)	C(46)—C(49)	1.521 (13)
O(1)—C(45)	1.225 (11)	C(50)—C(51)	1.534 (15)
O(2)—C(43)	1.228 (12)	C(50)—C(52)	1.528 (16)
O(3)—C(56)	1.272 (21)	C(50)—C(53)	1.550 (13)
O(3)—C(57)	1.402 (25)	C(55)—C(56)	1.426 (32)
N(1)—C(54)	1.158 (12)	C(57)—C(58)	1.341 (22)
C(41)—C(42)	1.451 (13)		

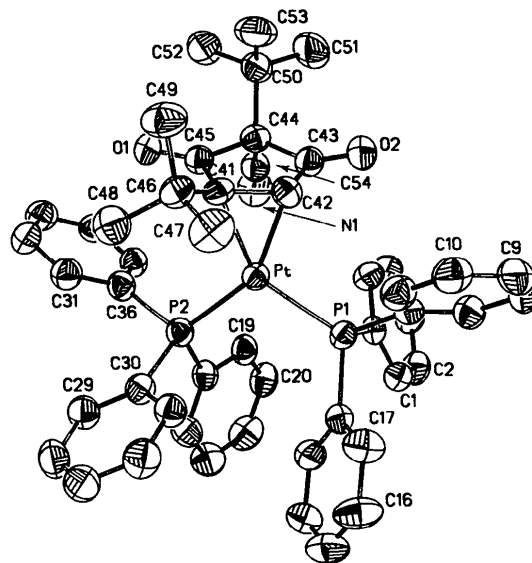
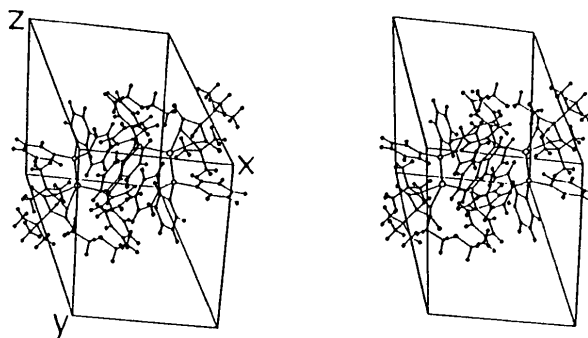
Fig. 1. The molecular structure for $[\text{Pt}(\text{C}_{14}\text{H}_{19}\text{NO}_2)\{\text{P}(\text{C}_6\text{H}_5)_3\}_2] \cdot \text{C}_4\text{H}_{10}\text{O}$ with the atom-labeling scheme.

Fig. 2. Unit-cell packing diagram.

from hexane (0.30 \times 0.21 \times 0.13 mm); Nicolet *R3m* diffractometer with graphite monochromator; ω scans; lattice parameters from least-squares fit of 25 reflections ($30 \leq 2\theta \leq 50^\circ$); empirical absorption correction ($\mu = 31.9 \text{ cm}^{-1}$, $T_{\text{max}}/T_{\text{min}} = 3.75$); $2\theta_{\text{max}} =$

48° ($h = \pm 13$, $k = \pm 16$, $l = +18$); $\bar{3}11$, 271, 148 standard reflections for 6460 reflections collected with 3% decay, 6192 unique ($R_{\text{int}} = 2.11\%$), 5014 observed with $F_o > 5\sigma(F_o)$, 1178 unobserved reflections. Direct-methods (*SOLV*) structure solution;

Table 3. Bond angles (°)

Pt(1)—Pt—Pt(2)	106.6 (1)	Pt—C(41)—C(46)	120.5 (5)
P(1)—Pt—C(41)	141.9 (2)	C(42)—C(41)—C(46)	122.1 (7)
P(2)—Pt—C(41)	111.1 (2)	C(45)—C(41)—C(46)	122.4 (7)
P(1)—Pt—C(42)	103.4 (2)	Pt—C(42)—C(41)	72.1 (5)
P(2)—Pt—C(42)	150.0 (2)	Pt—C(42)—C(43)	101.2 (5)
C(41)—Pt—C(42)	39.5 (3)	C(41)—C(42)—C(43)	110.4 (7)
Pt—P(1)—C(6)	114.1 (2)	O(2)—C(43)—C(42)	127.2 (8)
Pt—P(1)—C(12)	113.9 (2)	O(2)—C(43)—C(44)	122.9 (7)
C(6)—P(1)—C(12)	103.0 (3)	C(42)—C(43)—C(44)	109.8 (8)
Pt—P(1)—C(18)	117.6 (2)	C(43)—C(44)—C(45)	101.3 (6)
C(6)—P(1)—C(18)	105.0 (2)	C(43)—C(44)—C(50)	112.6 (7)
C(12)—P(1)—C(18)	101.4 (3)	C(45)—C(44)—C(50)	112.6 (6)
Pt—P(2)—C(24)	115.2 (2)	C(43)—C(44)—C(54)	111.7 (7)
Pt—P(2)—C(30)	114.3 (2)	C(45)—C(44)—C(54)	110.7 (7)
C(24)—P(2)—C(30)	104.0 (2)	C(50)—C(44)—C(54)	107.9 (6)
Pt—P(2)—C(36)	114.7 (2)	O(1)—C(45)—C(41)	127.6 (8)
P(24)—P(2)—C(36)	101.9 (3)	O(1)—C(45)—C(44)	122.3 (7)
C(30)—OP(2)—C(36)	105.3 (3)	C(41)—C(45)—C(44)	110.0 (7)
C(56)—O(3)—C(57)	115.9 (17)	C(41)—C(46)—C(47)	111.0 (8)
P(1)—C(6)—C(5)	115.6 (2)	C(41)—C(46)—C(48)	111.5 (7)
P(1)—C(12)—C(7)	121.4 (2)	C(47)—C(46)—C(48)	107.4 (8)
P(1)—C(12)—C(11)	118.6 (2)	C(41)—C(46)—C(49)	108.5 (7)
P(1)—C(18)—C(13)	119.6 (2)	C(47)—C(46)—C(49)	109.9 (8)
P(1)—C(18)—C(17)	120.4 (2)	C(48)—C(46)—C(49)	108.5 (8)
P(2)—C(24)—C(19)	117.9 (2)	C(44)—C(50)—C(51)	111.2 (8)
P(2)—C(24)—C(23)	122.0 (2)	C(44)—C(50)—C(52)	111.5 (8)
P(2)—C(30)—C(25)	116.9 (2)	C(51)—C(50)—C(52)	106.8 (8)
P(2)—C(30)—C(29)	123.1 (2)	C(44)—C(50)—C(53)	108.6 (7)
P(2)—C(36)—C(31)	122.2 (2)	C(51)—C(50)—C(53)	109.6 (9)
P(2)—C(36)—C(35)	117.8 (2)	C(52)—C(50)—C(53)	109.0 (8)
Pt—C(41)—C(42)	68.4 (4)	N(1)—C(54)—C(44)	179.2 (10)
Pt—C(41)—C(45)	101.6 (5)	O(3)—C(56)—C(55)	114.9 (20)
C(42)—C(41)—C(45)	108.5 (7)	O(3)—C(57)—C(58)	112.3 (17)

least-squares refinement on 479 parameters; all non-H atoms anisotropic, H atoms idealized and updated (C—H = 0.96 Å, $U = 1.2U$ of attached C),

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Carbonyl(2-cyano-3,3-dimethylbutanoic acid-*N*)(η^5 -cyclopentadienyl)-(triphenylphosphine)iron(I) Tetrafluoroborate

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Abstract. [Fe(C₅H₅)(CO)(C₇H₁₁NO₂){P(C₆H₅)₃]-[BF₄], $M_r = 627.2$, triclinic, $P\bar{1}$, $a = 11.123$ (5), $b = 12.085$ (6), $c = 12.855$ (6) Å, $\alpha = 101.91$ (4), $\beta = 93.67$ (4), $\gamma = 108.43$ (3)°, $V = 1588.4$ (12) Å³, $Z = 2$, $D_x = 1.311$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 5.71$ cm⁻¹, $F(000) = 648$, $T = 296$ K, $R_F = 5.29\%$ for

phenyl rings constrained to rigid hexagons (C—C = 1.395 Å). $R_F = 4.11\%$, $wR_F = 5.00\%$, $S = 1.072$, $w^{-1} = \sigma^2(F_o) + gF_o^2$, $g = 0.001$; $(\Delta/\sigma)_{\text{max}} = 0.18$; $\Delta\rho_{\text{max}} = 1.075$, $\Delta\rho_{\text{min}} = -0.407$ e Å⁻³; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); *SHELXTL* computer program (Sheldrick, 1983).

Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1. Bond lengths are given in Table 2 and angles are given in Table 3. Fig. 1 shows the labeled molecular structure of the compound and Fig. 2 shows the unit-cell packing diagram.*

Related literature. To our knowledge, no other structures of cyclopentenedione platinum diphosphine complexes have been published.

* Lists of structure factors, anisotropic thermal parameters and full lists of bond lengths have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52368 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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