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Acta Cryst. (1987). **C43**, 1253–1255

cis-(*E*)-Bis(7-methyl-1-naphthyl)bis(triphenylphosphine)platinum(II)

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(Received 23 June 1986; accepted 17 February 1987)

Abstract. $[\text{Pt}(\text{C}_{11}\text{H}_9)_2(\text{C}_{18}\text{H}_{15}\text{P}_2)] \cdot \text{CH}_2\text{Cl}_2$, $M_r = 1087.08$, monoclinic, space group $P2_1/a$, $a = 18.104$ (4), $b = 20.872$ (4), $c = 13.680$ (3) Å, $\beta = 106.27$ (4)°, $V = 4962.1$ Å³, $Z = 4$, $D_x = 1.455$, $D_m = 1.468$ g cm⁻³ (flotation method in CCl_4 /hexane), graphite-monochromated Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 30.65$ cm⁻¹, $F(000) = 2184$, $T = 295$ K, 6082 reflections measured, 3133 of which were taken as observed; final R value 0.049. Each molecule of the title compound crystallizes with one molecule of CH_2Cl_2 . The carbon atoms C(1) and C(12) of the naphthyl ligands and the P atoms of the phosphine ligands lie in a plane containing nearly dsp^2 -hybridized Pt. The two naphthyl ligands are approximately perpendicular (88.8 and 87.2°) to this plane and *anti*-orientated to one another (*E* conformation).

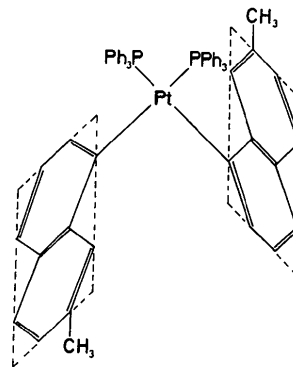


Fig. 1. Schematic illustration of *cis*-(*E*)-bis(7-methyl-1-naphthyl)-bis(triphenylphosphine)platinum(II).

Introduction. In the course of experiments on the stereochemistry of platinum–organic compounds we were interested in the mutual orientation of large planar aryl ligands directly bonded to platinum in *cis*- and *trans*-bis(aryl)bis(ligand)platinum(II) compounds (Brune, Wiege & Debaerdemaeker, 1984; Debaerdemaeker, Stapp & Brune, 1987; Debaerdemaeker, Weisemann & Brune, 1987). As a contribution to the stereochemistry of these compounds the structure of *cis*-(*E*)-bis(7-methyl-1-naphthyl)bis(triphenylphosphine)platinum(II) (Fig. 1) is reported here.

Experimental. The synthesis of the title compound was previously reported (Brune, Schäfer, Spohn & Weisemann, 1986). Crystals (0.3 × 0.1 × 0.1 mm approximately) were obtained by the very slow cooling of solutions of the title compound in CH_2Cl_2 /methanol (1/1). Philips PW-1100 four-circle diffractometer; Mo $K\alpha$ radiation, graphite monochromator, $\lambda = 0.71069$ Å. θ – 2θ scan; 6082 unique reflections measured, 3133 observed reflections [$F > 2.0\sigma(F)$, $-18 \leq h \leq 18$, $0 \leq k \leq 20$, $0 \leq l \leq 13$, $\theta_{\max} = 22^\circ$]. The intensities of three monitor reflections did not vary

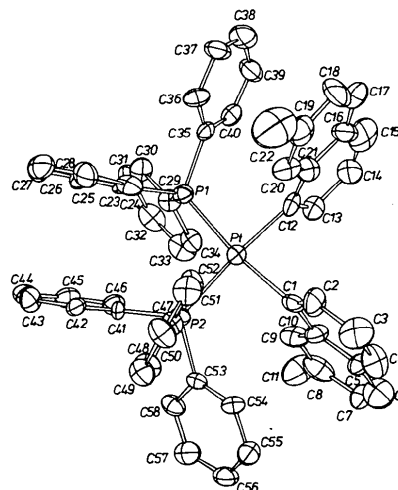
Table 1. Atomic coordinates for non-hydrogen atoms and temperature factors

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
Pt	0.2669 (1)	0.3978 (1)	0.7271 (1)	0.049 (1)
P(1)	0.2900 (2)	0.3214 (2)	0.6155 (3)	0.052 (4)
P(2)	0.3012 (2)	0.4906 (2)	0.6543 (3)	0.048 (4)
C(1)	0.2406 (9)	0.4549 (7)	0.8378 (13)	0.059 (19)
C(2)	0.2944 (12)	0.4670 (10)	0.9321 (14)	0.090 (26)
C(3)	0.2784 (12)	0.5030 (12)	1.0136 (19)	0.119 (33)
C(4)	0.2067 (13)	0.5218 (11)	0.9952 (14)	0.103 (30)
C(5)	0.1466 (12)	0.5114 (8)	0.9093 (18)	0.086 (26)
C(6)	0.0718 (14)	0.5326 (12)	0.8930 (21)	0.117 (35)
C(7)	0.0148 (14)	0.5242 (10)	0.8050 (19)	0.102 (31)
C(8)	0.0297 (15)	0.4902 (10)	0.7186 (19)	0.107 (31)
C(9)	0.1063 (10)	0.4661 (8)	0.7270 (17)	0.079 (24)
C(10)	0.1631 (11)	0.4758 (8)	0.8255 (16)	0.073 (23)
C(11)	-0.0271 (15)	0.4794 (13)	0.6222 (21)	0.135 (38)
C(12)	0.2401 (11)	0.3213 (9)	0.8081 (13)	0.071 (23)
C(13)	0.1576 (12)	0.3032 (9)	0.7811 (15)	0.096 (27)
C(14)	0.1359 (16)	0.2466 (10)	0.8316 (18)	0.129 (37)
C(15)	0.1891 (20)	0.2109 (12)	0.8985 (19)	0.145 (42)
C(16)	0.2701 (18)	0.2313 (10)	0.9319 (17)	0.099 (31)
C(17)	0.3333 (24)	0.1995 (15)	1.0022 (24)	0.136 (47)
C(18)	0.4061 (24)	0.2185 (15)	1.0279 (21)	0.139 (49)
C(19)	0.4264 (16)	0.2761 (14)	0.9818 (17)	0.110 (35)
C(20)	0.3716 (11)	0.3108 (9)	0.9098 (12)	0.082 (22)
C(21)	0.2921 (12)	0.2886 (9)	0.8816 (13)	0.084 (24)
C(22)	0.5101 (25)	0.2973 (19)	1.0024 (25)	0.214 (63)
C(23)	0.3734 (9)	0.3351 (7)	0.5643 (15)	0.058 (20)
C(24)	0.4413 (9)	0.3526 (8)	0.6438 (15)	0.068 (21)
C(25)	0.5113 (11)	0.3584 (10)	0.6160 (19)	0.091 (28)
C(26)	0.5126 (14)	0.3479 (10)	0.5181 (24)	0.097 (33)
C(27)	0.4477 (15)	0.3322 (10)	0.4438 (19)	0.095 (30)
C(28)	0.3759 (11)	0.3239 (8)	0.4654 (15)	0.073 (23)
C(29)	0.2069 (8)	0.3065 (7)	0.5060 (12)	0.054 (17)
C(30)	0.2064 (10)	0.2627 (8)	0.4316 (13)	0.068 (21)
C(31)	0.1415 (12)	0.2556 (9)	0.3452 (15)	0.087 (25)
C(32)	0.0740 (11)	0.2933 (10)	0.3401 (15)	0.090 (25)
C(33)	0.0735 (10)	0.3360 (10)	0.4191 (16)	0.087 (25)
C(34)	0.1405 (8)	0.3443 (8)	0.5021 (12)	0.060 (19)
C(35)	0.3083 (10)	0.2394 (7)	0.6649 (12)	0.057 (19)
C(36)	0.3852 (9)	0.2160 (8)	0.7095 (13)	0.066 (21)
C(37)	0.3950 (13)	0.1535 (8)	0.7513 (13)	0.094 (26)
C(38)	0.3293 (14)	0.1139 (9)	0.7403 (15)	0.098 (27)
C(39)	0.2533 (13)	0.1366 (9)	0.6951 (14)	0.080 (25)
C(40)	0.2459 (10)	0.1980 (9)	0.6572 (13)	0.067 (21)
C(41)	0.3101 (9)	0.4855 (6)	0.5254 (10)	0.045 (16)
C(42)	0.3821 (9)	0.4999 (7)	0.5035 (11)	0.051 (17)
C(43)	0.3841 (11)	0.4919 (8)	0.4021 (13)	0.067 (21)
C(44)	0.3221 (12)	0.4694 (8)	0.3257 (14)	0.079 (23)
C(45)	0.2527 (10)	0.4546 (8)	0.3503 (13)	0.066 (20)
C(46)	0.2459 (10)	0.4613 (7)	0.4500 (13)	0.061 (19)
C(47)	0.3945 (9)	0.5229 (7)	0.7303 (11)	0.053 (18)
C(48)	0.4169 (9)	0.5845 (7)	0.7112 (14)	0.067 (21)
C(49)	0.4868 (10)	0.6095 (10)	0.7700 (13)	0.079 (21)
C(50)	0.5362 (10)	0.5710 (9)	0.8497 (15)	0.086 (23)
C(51)	0.5130 (9)	0.5104 (9)	0.8672 (14)	0.080 (23)
C(52)	0.4412 (9)	0.4835 (9)	0.8055 (12)	0.064 (20)
C(53)	0.2398 (8)	0.5604 (6)	0.6502 (11)	0.045 (16)
C(54)	0.2429 (10)	0.5922 (7)	0.7385 (13)	0.068 (21)
C(55)	0.1966 (12)	0.6453 (8)	0.7415 (15)	0.093 (26)
C(56)	0.1437 (11)	0.6659 (8)	0.6485 (17)	0.095 (27)
C(57)	0.1421 (9)	0.6360 (8)	0.5570 (15)	0.079 (22)
C(58)	0.1898 (9)	0.5825 (7)	0.5579 (13)	0.063 (20)
C(59)	0.1915 (14)	0.3523 (12)	0.0516 (19)	0.124 (35)
Cl(1)	0.2679 (7)	0.3268 (4)	0.1579 (7)	0.210 (17)
Cl(2)	0.1138 (6)	0.3711 (6)	0.0853 (9)	0.246 (21)

Table 2. Main bond distances (Å) and angles (°) with e.s.d.'s in parentheses

Pt—P(1)	2.32 (1)	Pt—C(1)	2.08 (2)
Pt—P(2)	2.34 (1)	Pt—C(12)	2.08 (2)
P(1)—Pt—P(2)	99.8 (2)	P(1)—Pt—C(12)	86.3 (6)
C(1)—Pt—C(12)	85.2 (7)	P(2)—Pt—C(1)	88.7 (5)

Fig. 2. Projection of the molecule approximately perpendicular to the plane defined by the four valences of the dsp^2 -hybridized Pt atom.*

squares on F , first with isotropic and then with anisotropic temperature parameters, using unit weights (Sheldrick, 1976) until the parameter shifts were less than the corresponding standard deviations. Final $R = 0.049$ for 3133 reflections. $\Delta/\sigma \leq 0.1$, maximum peak in final difference map 1.1 e \AA^{-3} . Scattering factors of Cromer & Mann (1968) and Doyle & Turner (1968) were used. The *SHELX76* (Sheldrick, 1976) system was used for all computations. No corrections for secondary extinction.

Discussion. Final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, main interatomic distances and angles in Table 2.* A view of the molecule and the atomic numbering are depicted in Fig. 2.

The structure exhibits the following characteristics:

(1) Pt, P(1), P(2) and the two C atoms bonded directly to Pt [C(1) and C(12)] according to the continuous numbering in Fig. 2] lie in a plane; hence the

during the period of measurement. The cell parameters were obtained from the refinement of 25 reflections ($10 < \theta < 18^\circ$). Empirical absorption correction (Walker & Stuart, 1983), min. and max. absorption coefficients 0.964 and 1.135. The location of the Pt atom was found by a Patterson synthesis. The remaining atoms (except for H atoms, which were not located) were found in successive difference Fourier maps. The structure was refined by full-matrix least

* Lists of structure factors, anisotropic thermal parameters and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43797 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

central atom is dsp^2 -hybridized. The distances of Pt, P(1), P(2), C(1) and C(12) from the least-squares plane are given in Table 3. As a consequence of the large van der Waals volume of the triphenylphosphine ligands the angle P(1)–Pt–P(2) is 99.8° and is markedly enlarged with respect to the ideal dsp^2 angle of 90° . The two Pt–P bond lengths are equal within the standard deviations as are the Pt–C bond lengths. The significant difference between the two Pt–C bond lengths (a phenomenon as yet unexplained) observed in the structurally related compound (2,3,5,6- η^4 -bicyclo[2.2.1]hepta-2,5-diene)bis(2-ethoxy-1-naphthyl)-platinum(II) [Pt–C bond distances 2.01 and 2.08 Å (Debaerdemaeker, Weisemann & Brune, 1987)] is not found here. Instead, the lengths of the Pt–P and Pt–C bonds are approximately equal to those measured in compounds of the type *cis*- and *trans*-bis(phenyl)-bis(triphenylphosphine)platinum(II) (see Table 4) and are thus normal.

(2) The two naphthyl ligands are planar and orientate their planes nearly perpendicularly (88.8 and 87.2°) to the plane defined by the four valences of the dsp^2 -hybridized Pt. The 1-naphthyl ring systems are mutually *anti*-orientated (*E* conformation).

(3) The C atoms of the methyl groups lie in the plane defined by the ten C atoms of the naphthalene rings (deviations from the least-squares plane: 0.05 and 0.08 Å).

(4) The Pt atom, however, is significantly pushed out of the naphthalene planes (by 0.18 and 0.22 Å respectively). This might be ascribed to the steric strain caused by *peri* interaction of the Pt atom with the H atoms bound to C(10) and C(21) respectively.

We thank the Verband der Chemischen Industrie, Fonds der Chemischen Industrie, for financial support.

Acta Cryst. (1987). C43, 1255–1258

Structure du Complexe Bis(oxyde de triphénylphosphine)tétrakis(tétrahydroborato)uranium(IV)

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(Reçu le 22 septembre 1986, accepté le 23 février 1987)

Abstract. $[U(BH_4)_4(C_{18}H_{15}OP)_2]$, $M_r = 853.98$, monoclinic, $P2_1$, $a = 10.221(2)$, $b = 16.027(4)$, $c = 11.911(4)$ Å, $\beta = 99.05(2)^\circ$, $V = 1927(2)$ Å³, $Z = 2$, $D_x = 1.472$ Mg m^{−3}, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 4.10$ mm^{−1}, $F(000) = 840$, $T = 295$ K, $R = 0.040$ for

0108-2701/87/071255-04\$01.50

Table 3. Distances of the Pt atom and of the atoms directly bonded to Pt from the least-squares plane (Å, e.s.d.'s in parentheses)

Pt	−0.02 (1)	C(1)	−0.03 (1)
P(1)	−0.03 (1)	C(12)	0.04 (1)
P(2)	0.04 (1)		

Table 4. Pt–P and Pt–C bond distances (Å) in compounds of the type *cis*- and *trans*-bis(phenyl)-bis(triphenylphosphine)platinum(II)

Compound*	Pt–P	Pt–C	Reference
1	2.31	2.03	Debaerdemaeker, Klein, Wiege & Brune (1981)
2	2.30	2.10	Brune, Wiege & Debaerdemaeker (1984)
3	2.33	2.06	Brune, Wiege & Debaerdemaeker (1984)
4	2.30	2.08	Ertl, Debaerdemaeker & Brune (1982)

* (1): *cis*-Bis(2-methoxyphenyl)bis(triphenylphosphine)platinum(II), (2): *trans*-bis(2,4,6-trimethoxyphenyl)bis(triphenylphosphine)platinum(II), (3): *cis*-bis(2-nitrophenyl)bis(triphenylphosphine)platinum(II), (4): *trans*-di-phenylbis(triphenylphosphine)platinum(II).

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1320 independent reflections. The structure is of molecular type. The U atom is hexacoordinated by two triphenylphosphine oxide ligands in *trans* positions [U–O 2.33(2) and 2.24(2) Å] and four tetrahydroborate ions. The significantly different U–B

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