Barclay, J. E., Evans, D. J., Hughes, D. L. & Leigh, G. J. (1993). *J. Chem. Soc. Dalton Trans.* pp. 69–73.
Bierbach, U., Saak, W., Haase, D. & Pohl, S. (1990). *Z. Naturforsch. Teil B*, **45**, 45–52.
Desai, J. U., Gordon, J. C., Kraatz, H.-B., Lee, V. T., Owens-Waltermire, B. E., Poli, R., Rheingold, A. L. & White, C. B. (1994). *Inorg. Chem.* **33**, 3752–3769.
Dunitz, J. D. & Seiler, P. (1973). *Acta Cryst.* B**29**, 589–595.
Enraf–Nonius (1977). *CAD-4 Operations Manual.* Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf–Nonius, Delft, The Netherlands.
Falvello, L. R., Mattamana, S. P. & Poli, R. (1995). *Acta Cryst.* C**51**, 569–571.
Green, J. C., Green, M. L. H., Mountford, P. & Parkington, M. J. (1990). *J. Chem. Soc. Dalton Trans.* p. 3407.
Ibers, J. A. & Hamilton, W. C. (1964). *Acta Cryst.* **17**, 781.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Killean, R. C. G. & Lawrence, J. L. (1969). *Acta Cryst.* B**25**, 1750–1752.
Lane, H. P., Godfrey, S. M., McAuliffe, C. A. & Pritchard, R. G. (1994). *J. Chem. Soc. Dalton Trans.* pp. 3249–3256.
Pasynskii, A. A., Eremenko, I. L., Stomakhina, E. E., Nefedov, S. E., Ellert, O. G., Yanovsky, A. I. & Struchkov, Y. T. (1991). *J. Organomet. Chem.* **406**, 383–390.
Pohl, S. & Saak, W. (1985). *Z. Anorg. Allg. Chem.* **523**, 25–32.
Saak, W., Haase, D. & Pohl, S. (1988). *Z. Naturforsch. Teil B*, **43**, 289–294.
Saak, W. & Pohl, S. (1987). *Z. Anorg. Allg. Chem.* **552**, 186–194.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* University of Göttingen, Germany.
Shin, J. H. & Parkin, G. (1994). *Polyhedron*, **13**, 1489–1493.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.
Zandbergen, H. W., Verschoor, G. C. & Ijdo, D. J. W. (1979). *Acta Cryst.* B**35**, 1425–1427.

Acta Cryst. (1996). C**52**, 1101-1104

Two Forms of Bromo(cycloheptatrienylidene)bis(triphenylphosphine)platinum(II)

Jerzy Klosin, William M. Jones and Khalil A. Abboud*

Department of Chemistry, University of Florida, PO Box 117200, Gainesville, FL 32611-7200, USA

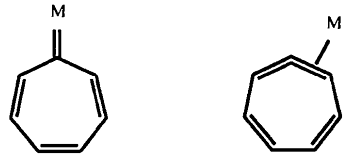
(*Received 27 May 1994; accepted 24 July 1995*)

Abstract

The title complex crystallizes in two different crystal systems: one form in the monoclinic system, as $[PtBr(C_7H_6)(C_{18}H_{15}P)_2]BF_4.CD_2Cl_2.C_4D_8O$, and the other in the orthorhombic crystal system, as $[PtBr(C_7H_6)(C_{18}H_{15}P)_2]BF_4.C_4H_8O$. The crystal structure determination of the complex bromo(cycloheptatrienylidene)bis(triphenylphosphine)platinum(II), $[PtBr(C_7H_6)(C_{18}H_{15}P)_2]$, revealed that the Pt^{II} cation is coordinated to a Br atom, two triphenylphosphine donors and cycloheptatrienylidene. The complex has square-planar geometry around Pt with the triphenylphosphine ligands being *trans* to each other. The plane of the cycloheptatrienylidene ligand is almost perpendicular to the coordination plane.

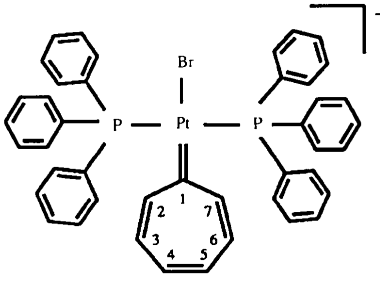
Comment

It has been shown that when monocyclic C_7H_6 is attached to a transition metal it can exist in two different forms, an allene or a carbene. The preference for one form over the other depends on the transition metal and its oxidation state (Winchester, 1985).



Carbene form Allene form

Several carbene complexes containing monocyclic C_7H_6 have been synthesized and two of them have been structurally characterized (Riley, Davis, Allison & Jones, 1980). The only transition metal for which both forms have been observed is platinum. The Pt^0 complex was found to be exclusively in the allene form both in solution (Winchester & Jones, 1985) and in the crystal (Abboud, Lu & Jones, 1992). Recently, a Pt^{II} complex containing monocyclic C_7H_6 was synthesized and was shown spectroscopically to exist in carbene form in solution (Lu, Jones & Winchester, 1993). This study confirms that the carbene form is also preferred in the crystalline state. Suitable light-yellow crystals of (I) were obtained from a solution mixture of deuterized methylene chloride and thf-d^8. Initial attempts at crystallizing the Pt^{II} complex yielded light-yellow crystals from a mixture of methylene chloride and thf. After several recrystallizations to obtain better crystals, data were collected on a small crystal and the structure, (II), was found to be in the orthorhombic system. The change in the solvent mixture from methylene chloride and thf to deuterized methylene chloride and thf-d^8 caused a change in the crystal system from orthorhombic ($P2_12_12_1$) to monoclinic ($P2_1/n$).



$BF_4^-.CD_2Cl_2.C_4D_8O$ (I)
$BF_4^-.C_4H_8O$ (II)

A displacement ellipsoid drawing (*SHELXTL-Plus*; Sheldrick, 1991) of the Pt^{II} complex in (I) with the atom-labelling scheme is given in Fig. 1. The coordination geometry around Pt^{II} in (I) is square-planar with a maximum deviation from the mean plane of Br, P1, P2, Pt and C1 of 0.02 (1) Å for the P atoms. This deviation is substantially larger in (II) and is equal to 0.19 Å for C1 (average deviation is 0.03 Å). The difference between the Br—Pt—C1 angles in (I) and (II) [179.7 (5) and 169.8 (5)°, respectively] also indicates larger distortions in (II). Another significant difference between (I) and (II) is the position of the plane of the seven-membered ring relative to the plane of Br, P1, P2, Pt and C1. In (I) the dihedral angle between these planes is equal to 103.5 (3)°, whereas in (II) it is 94.6 (5)°. These angles agree with observed values in several other Pt^{II}–carbene complexes (Shubert, 1983; Michelin, Zanotto, Braga, Sabatino & Angelici, 1988; Garlaschelli, Malatesta, Panzeri, Albinati & Ganazzoli, 1987). The remaining geometrical features of both complexes are the same or very similar. The seven-membered ring is planar in both compounds. The Pt—C1 (carbene) bond lengths of 1.99 (1) and 2.03 (2) Å in (I) and (II), respectively, are in good agreement with each other and with corresponding distances in other square-planar Pt^{II}–carbene complexes (Shubert, 1983; Michelin *et al.*, 1988; Garlaschelli *et al.*, 1987). The Pt—Br bond length of 2.513 (1) Å is slightly lengthened compared with those of other Pt^{II}–carbene complexes with the Pt—Br bond *trans* to the carbene (Michelin *et al.*, 1988).

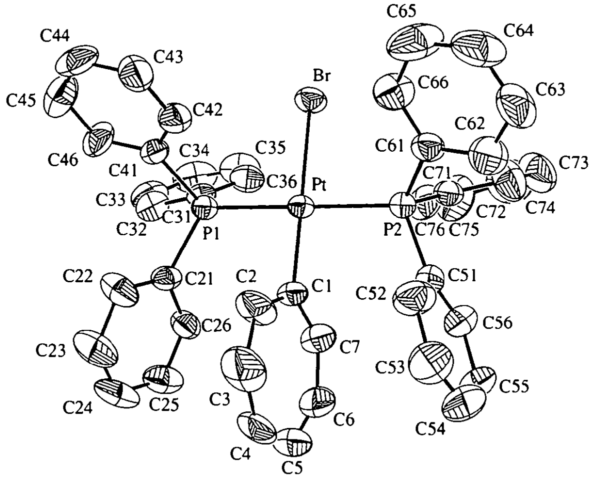


Fig. 1. Molecular structure of the Pt^{II} complex of compound (I), with 50% probability ellipsoids and showing the atom-numbering scheme.

Experimental

The title compounds were prepared according to a published procedure (Lu *et al.*, 1993).

Compound (I)

Crystal data

$[PtBr(C_7H_6)(C_{18}H_{15}P)_2]$-
$BF_4.CD_2Cl_2.C_4D_8O$
$M_r = 1143.5$
Monoclinic
$P2_1/n$
$a = 11.360 (2)$ Å
$b = 35.845 (5)$ Å
$c = 11.495 (2)$ Å
$\beta = 90.95 (2)°$
$V = 4680 (1)$ Å^3
$Z = 4$
$D_x = 1.623$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 10–11°$
$\mu = 4.09$ mm^{-1}
$T = 293$ K
Block
0.57 × 0.48 × 0.28 mm
Yellow

Data collection

Seimens *P*3*m*/*V* diffractometer
ω scans
Absorption correction: analytical
$T_{min} = 0.253$, $T_{max} = 0.409$
8618 measured reflections
8034 independent reflections
5090 observed reflections $[F > 4\sigma(F)]$

$R_{int} = 0.0146$
$\theta_{max} = 25°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 42$
$l = -13 \rightarrow 13$
4 standard reflections monitored every 100 reflections
intensity decay: 4%

Refinement

Refinement on *F*
$R = 0.0509$
$wR = 0.0578$
$S = 1.57$
5090 reflections
482 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0004F]^2$

$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 1.1$ e Å^{-3}
$\Delta\rho_{min} = -0.9$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (II)

Crystal data

$[PtBr(C_7H_6)(C_{18}H_{15}P)_2]$-
$BF_4.C_4H_8O$
$M_r = 1048.57$
Orthorhombic
$P2_12_12_1$
$a = 10.943 (3)$ Å
$b = 15.861 (4)$ Å
$c = 27.756 (7)$ Å
$V = 4817 (2)$ Å^3
$Z = 4$
$D_x = 1.446$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 10–11°$
$\mu = 3.86$ mm^{-1}
$T = 293$ K
Block
0.72 × 0.23 × 0.2 mm
Yellow

Data collection

Seimens *P*3*m*/*V* diffractometer

$R_{int} = 0.0577$
$\theta_{max} = 25°$

ω scans
Absorption correction:
analytical
$T_{\min} = 0.451$, $T_{\max} = 0.512$
5877 measured reflections
4731 independent reflections
3111 observed reflections
$[F > 6\sigma(F)]$

$h = 0 \rightarrow 13$
$k = 0 \rightarrow 18$
$l = -32 \rightarrow 32$
4 standard reflections
monitored every 100 reflections
intensity decay: 4%

Refinement

Refinement on F
$R = 0.0646$
$wR = 0.0736$
$S = 2.05$
3111 reflections
261 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0004F]^2$

$(\Delta/\sigma)_{\max} = 2.794$
$\Delta\rho_{\max} = 1.5$ e Å^{-3}
$\Delta\rho_{\min} = -1$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (I)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Pt	0.22119 (4)	0.135860 (10)	0.04144 (4)	0.04120 (14)
Br	0.20729 (12)	0.18101 (3)	−0.12543 (10)	0.0596 (5)
P1	0.2376 (3)	0.08695 (8)	−0.0914 (3)	0.0435 (10)
P2	0.2095 (3)	0.18529 (8)	0.1718 (3)	0.0480 (11)
C1	0.2313 (13)	0.1000 (3)	0.1734 (10)	0.057 (5)
C2	0.129 (2)	0.0779 (4)	0.1960 (12)	0.085 (7)
C3	0.118 (2)	0.0517 (5)	0.282 (2)	0.121 (9)
C4	0.183 (3)	0.0375 (5)	0.363 (2)	0.16 (2)
C5	0.303 (3)	0.0475 (6)	0.385 (2)	0.17 (2)
C6	0.371 (2)	0.0727 (5)	0.327 (2)	0.131 (10)
C7	0.3391 (15)	0.0963 (4)	0.2353 (11)	0.085 (7)
C21	0.2536 (11)	0.0416 (3)	−0.0246 (10)	0.053 (5)
C22	0.1628 (13)	0.0161 (4)	−0.0188 (13)	0.080 (6)
C23	0.179 (2)	−0.0163 (4)	0.0430 (14)	0.101 (8)
C24	0.281 (2)	−0.0247 (4)	0.0977 (14)	0.0974 (7)
C25	0.3730 (14)	0.0007 (4)	0.0932 (12)	0.081 (6)
C26	0.3565 (12)	0.0326 (3)	0.0334 (11)	0.062 (5)
C31	0.3663 (10)	0.0891 (3)	−0.1858 (10)	0.049 (4)
C32	0.3965 (11)	0.0582 (4)	−0.2505 (11)	0.064 (5)
C33	0.4886 (15)	0.0591 (5)	−0.3260 (13)	0.087 (7)
C34	0.5547 (14)	0.0923 (6)	−0.3322 (14)	0.095 (8)
C35	0.5276 (12)	0.1212 (5)	−0.2655 (14)	0.088 (7)
C36	0.4360 (12)	0.1206 (4)	−0.1892 (12)	0.071 (6)
C41	0.1066 (10)	0.0830 (3)	−0.1843 (9)	0.047 (4)
C42	0.1011 (12)	0.0590 (4)	−0.2790 (12)	0.079 (6)
C43	0.002 (2)	0.0571 (5)	−0.3455 (13)	0.094 (8)
C44	−0.0935 (15)	0.0783 (5)	−0.3211 (14)	0.094 (8)
C45	−0.0885 (14)	0.1019 (4)	−0.225 (2)	0.086 (7)
C46	0.0109 (12)	0.1050 (3)	−0.1602 (12)	0.068 (5)
C51	0.2158 (13)	0.1705 (3)	0.3230 (10)	0.058 (5)
C52	0.116 (2)	0.1545 (4)	0.3697 (12)	0.090 (7)
C53	0.119 (2)	0.1398 (5)	0.4835 (14)	0.109 (9)
C54	0.216 (2)	0.1423 (6)	0.549 (2)	0.128 (11)
C55	0.313 (2)	0.1576 (5)	0.5059 (15)	0.114 (10)
C56	0.315 (2)	0.1723 (4)	0.3889 (12)	0.088 (7)
C61	0.0807 (12)	0.2137 (3)	0.1657 (11)	0.060 (5)
C62	0.0064 (12)	0.2158 (4)	0.0707 (13)	0.080 (6)
C63	−0.092 (2)	0.2379 (6)	0.0682 (15)	0.124 (9)
C64	−0.115 (2)	0.2609 (5)	0.163 (2)	0.109 (9)
C65	−0.045 (2)	0.2602 (4)	0.260 (2)	0.098 (8)
C66	0.051 (2)	0.2359 (4)	0.2625 (13)	0.096 (7)
C71	0.3341 (12)	0.2164 (3)	0.1569 (11)	0.058 (5)
C72	0.4336 (14)	0.2046 (4)	0.1002 (12)	0.082 (6)
C73	0.533 (2)	0.2259 (6)	0.092 (2)	0.119 (9)
C74	0.531 (2)	0.2614 (7)	0.136 (2)	0.139 (12)
C75	0.438 (2)	0.2737 (5)	0.194 (2)	0.127 (11)
C76	0.339 (2)	0.2512 (4)	0.209 (2)	0.101 (8)
C80	0.731 (2)	0.1075 (6)	0.132 (2)	0.175 (13)
Cl1	0.8511 (7)	0.1336 (3)	0.1082 (7)	0.227 (5)
Cl2	0.6186 (6)	0.1159 (2)	0.0398 (7)	0.198 (4)
C1′ †	0.758 (2)	0.1613 (7)	0.500 (2)	0.155 (8)‡
C2′ †	0.791 (4)	0.1871 (12)	0.394 (4)	0.32 (2)‡
C3′ †	0.667 (3)	0.2089 (8)	0.362 (2)	0.179 (10)‡
C4′ †	0.629 (3)	0.1698 (11)	0.362 (3)	0.27 (2)‡
C5′ †	0.648 (3)	0.1889 (11)	0.502 (3)	0.27 (2)‡
B	0.7293 (8)	0.0376 (2)	0.3804 (8)	0.27 (2)‡
F1	0.7143 (8)	0.0324 (2)	0.2645 (8)	0.228 (7)‡
F2	0.7327 (8)	0.0742 (2)	0.4035 (8)	0.258 (8)‡
F3	0.8306 (8)	0.0217 (2)	0.4156 (8)	0.245 (7)‡
F4	0.6395 (8)	0.0219 (2)	0.4368 (8)	0.322 (10)‡

† thf sites, refined isotropically as a cyclopentane molecule (see below).
‡ $U_{\rm iso}$ (see below).

Table 2. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (II)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Pt	1.53790 (8)	0.74033 (5)	0.40212 (3)	0.0397 (2)†
Br	1.7432 (2)	0.7448 (2)	0.44304 (8)	0.0549 (8)†
P1	1.5402 (8)	0.8856 (3)	0.3916 (2)	0.045 (2)†
P2	1.5449 (7)	0.5954 (3)	0.4041 (3)	0.042 (2)†
C1	1.361 (2)	0.737 (2)	0.3813 (7)	0.044 (6)
C2	1.330 (2)	0.733 (2)	0.3316 (8)	0.057 (6)
C3	1.219 (2)	0.735 (2)	0.3097 (9)	0.069 (7)
C4	1.100 (3)	0.749 (2)	0.3290 (10)	0.089 (8)
C5	1.073 (3)	0.758 (2)	0.3787 (10)	0.095 (9)
C6	1.149 (2)	0.762 (2)	0.4196 (9)	0.077 (8)
C7	1.276 (2)	0.754 (2)	0.4183 (7)	0.055 (6)
C21	1.523 (2)	0.9437 (13)	0.4507 (8)	0.049 (6)
C22	1.522 (3)	0.898 (2)	0.4905 (10)	0.075 (8)
C23	1.503 (3)	0.946 (2)	0.5349 (12)	0.097 (11)
C24	1.474 (3)	1.034 (2)	0.5349 (11)	0.094 (10)
C25	1.463 (3)	1.076 (2)	0.4916 (11)	0.089 (9)
C26	1.485 (3)	1.033 (2)	0.4449 (10)	0.080 (9)
C31	1.684 (2)	0.921 (2)	0.3608 (9)	0.048 (7)
C32	1.710 (3)	1.008 (2)	0.3617 (11)	0.077 (9)
C33	1.804 (3)	1.036 (2)	0.3374 (11)	0.073 (9)
C34	1.874 (3)	0.967 (2)	0.3103 (13)	0.097 (11)
C35	1.850 (3)	0.892 (2)	0.3046 (11)	0.086 (10)
C36	1.745 (3)	0.866 (2)	0.3337 (11)	0.082 (9)
C41	1.427 (2)	0.9248 (15)	0.3535 (9)	0.050 (7)
C42	1.315 (3)	0.947 (2)	0.3689 (10)	0.078 (9)
C43	1.219 (4)	0.983 (3)	0.334 (2)	0.121 (14)
C44	1.245 (4)	0.986 (2)	0.2897 (14)	0.113 (13)
C45	1.357 (3)	0.952 (2)	0.2727 (12)	0.094 (10)
C46	1.447 (2)	0.9232 (14)	0.3039 (9)	0.054 (6)
C51	1.544 (3)	0.5621 (15)	0.4668 (9)	0.064 (7)
C52	1.630 (3)	0.503 (2)	0.4836 (9)	0.063 (7)
C53	1.623 (3)	0.478 (2)	0.5311 (10)	0.077 (9)
C54	1.519 (3)	0.506 (2)	0.5591 (10)	0.078 (9)
C55	1.443 (3)	0.561 (2)	0.5438 (10)	0.077 (9)
C56	1.450 (3)	0.590 (2)	0.4951 (10)	0.073 (8)
C61	1.416 (2)	0.5405 (14)	0.3725 (8)	0.040 (6)
C62	1.423 (2)	0.546 (2)	0.3243 (10)	0.061 (8)
C63	1.337 (3)	0.509 (2)	0.2987 (9)	0.062 (7)
C64	1.240 (3)	0.463 (2)	0.3176 (13)	0.094 (11)
C65	1.235 (4)	0.456 (2)	0.3705 (14)	0.110 (12)
C66	1.324 (4)	0.502 (2)	0.3943 (13)	0.109 (11)
C71	1.675 (3)	0.543 (2)	0.3744 (10)	0.057 (8)
C72	1.768 (3)	0.593 (2)	0.3530 (10)	0.064 (8)
C73	1.873 (3)	0.549 (2)	0.3301 (10)	0.071 (9)
C74	1.871 (2)	0.464 (2)	0.3319 (9)	0.053 (7)

C75	1.791 (3)	0.420 (2)	0.3507 (9)	0.060 (7)
C76	1.693 (2)	0.462 (2)	0.3733 (9)	0.055 (8)
O80‡	0.526 (4)	0.716 (2)	0.2373 (14)	0.087 (13)
C81‡	0.635 (6)	0.707 (3)	0.225 (2)	0.06 (2)
C82‡	0.622 (8)	0.756 (6)	0.171 (3)	0.13 (3)
C83‡	0.507 (5)	0.723 (4)	0.155 (2)	0.08 (2)
C84‡	0.479 (11)	0.693 (7)	0.193 (4)	0.11 (5)
O90§	0.245 (6)	0.750 (4)	0.045 (2)	0.18 (2)
C91§	0.142 (9)	0.705 (6)	0.044 (3)	0.17 (4)
C92§	0.165 (9)	0.661 (6)	−0.005 (3)	0.15 (3)
C93§	0.245 (6)	0.714 (4)	−0.028 (2)	0.10 (2)
C94§	0.305 (6)	0.768 (5)	0.015 (3)	0.11 (2)
B	1.0000 (12)	0.7225 (9)	0.1973 (5)	0.43 (7)
F1	1.1212 (12)	0.7330 (9)	0.1941 (5)	0.138 (7)
F2	0.9464 (12)	0.7972 (9)	0.2049 (5)	0.209 (12)
F3	0.9747 (12)	0.6706 (9)	0.2341 (5)	0.31 (2)
F4	0.9578 (12)	0.6890 (9)	0.1563 (5)	0.207 (12)

† U_{eq} (see below). ‡ Occupancy = 0.48 (1) (see below). § Occupancy = 0.52 (1) (see below).

Table 3. *Selected geometric parameters (Å, °) for (I) and (II)*

	(I)	(II)
Pt—Br	2.513 (1)	2.518 (2)
Pt—P1	2.334 (3)	2.323 (6)
Pt—P2	2.326 (3)	2.300 (5)
Pt—C1	1.99 (1)	2.03 (2)
P1—C21	1.80 (1)	1.89 (2)
P1—C31	1.84 (1)	1.88 (3)
P1—C41	1.82 (1)	1.74 (3)
P2—C51	1.82 (1)	1.82 (3)
P2—C61	1.78 (1)	1.87 (2)
P2—C71	1.81 (1)	1.84 (3)
C1—C2	1.43 (2)	1.42 (3)
C1—C7	1.41 (2)	1.41 (3)
C2—C3	1.37 (2)	1.36 (3)
C3—C4	1.28 (3)	1.42 (4)
C4—C5	1.43 (4)	1.42 (4)
C5—C6	1.37 (3)	1.41 (4)
C6—C7	1.40 (2)	1.39 (3)
Br—Pt—P1	89.4 (1)	91.1 (2)
Br—Pt—P2	89.9 (1)	89.3 (2)
P1—Pt—P2	178.4 (1)	173.6 (2)
P1—Pt—C1	90.5 (3)	89.9 (7)
P2—Pt—C1	90.3 (3)	90.9 (7)
C1—Pt—Br	179.7 (5)	169.8 (5)
C2—C1—C7	124 (1)	124 (2)
C2—C1—Pt	117 (1)	120 (1)
C7—C1—Pt	119 (1)	115 (1)
C3—C2—C1	126 (2)	130 (2)
C4—C3—C2	137 (2)	131 (2)
C5—C4—C3	124 (2)	125 (3)
C6—C5—C4	129 (2)	132 (3)
C7—C6—C5	128 (2)	124 (2)
C1—C7—C6	131 (2)	134 (2)

Crystals of both compounds lose transparency in the open air; thus data crystals were mounted in thin-walled glass capillaries to preserve them from solvent loss. Both structures were solved by the heavy-atom method and refined using full-matrix least-squares methods. In (I), all non-H atoms of the complex and the methylene Cl atoms were refined with anisotropic displacement parameters. The thf molecule was disordered, but could not be resolved. The position of the O atom in thf is indistinguishable because of the disorder; thus thf was refined as a cyclopentane molecule (its H atoms not included) with isotropic displacement parameters. The BF_4 molecule had all B—F bonds idealized and was refined as a rigid group with isotropic displacement parameters. All of the H atoms of (I) were placed in calculated idealized positions and their displacement parameters fixed. In (II), only Pt, Br, P1 and P2 were refined with anisotropic displacement parameters. Refinement of the remaining non-H atoms with anisotropic displacement parameters did not offer any improvement, and thus was not adopted. The two partial thf molecules were found in general positions. Their site-occupation factors were refined to 0.48 (1) for molecule O80–C84 and therefore molecule O90–C94 has a site-occupation factor of 0.52 (1). The thf molecules were refined with isotropic displacement parameters. BF_4 was treated in the same manner as in (I). The H atoms of (II) were not located from a difference Fourier map and were not placed in calculated idealized positions. They were not included in the refinement because all of the C atoms were refined only isotropically. The high $(\Delta/\sigma)_{max}$ value during the last cycle of refinement of (II) belongs to thf. No significant $(\Delta/\sigma)_{max}$ value was observed for any of the parameters of the Pt^{II} complex.

The ω-scan width was symmetrically over 1.2° about the $K\alpha_{1,2}$ maximum and the background was offset 1.0 and −1.0 in ω from the $K\alpha_{1,2}$ maximum. The scan speed was a variable 3–6° min^{-1} (depending upon intensity). The linear absorption coefficient was calculated using values from *International Tables for X-ray Crystallography* (1974). Programs used: *SHELXTL-Plus* (Sheldrick, 1991) for cell refinement, data collection, data reduction, structure solution (direct methods), and molecular graphics; *SHELX76* (Sheldrick, 1976) for structure refinement (full-matrix least-squares).

KAA wishes to acknowledge the University of Florida, Division of Sponsored Research, for financial support of the crystallography work. Acknowledgement is made to the National Science Foundation for the support of this research.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1018). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Abboud, K. A., Lu, Z. & Jones, W. M. (1992). *Acta Cryst.* **C48**, 909–913.

Garlaschelli, L., Malatesta, M. C., Panzeri, S., Albinati, A. & Ganazzoli, F. (1987). *Organometallics*, **6**, 63–72.

Lu, Z., Jones, W. M. & Winchester, W. R. (1993). *Organometallics*, **12**, 1344–1350.

Michelin, R. A., Zanotto, L., Braga, D., Sabatino, P. & Angelici, R. J. (1988). *Inorg. Chem.* **27**, 85–92; **27**, 93–99.

Riley, P. E., Davis, R. E., Allison, N. T. & Jones, W. M. (1980). *J. Am. Chem. Soc.* **102**, 2458–2460.

Schubert, U. (1983). *Transition Metal Carbene Complexes*, p. 73. Weinheim: Verlag Chemie.

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. University of Cambridge, England.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Winchester, W. R. (1985). PhD dissertation. University of Florida, USA.

Winchester, W. R. & Jones, W. M. (1985). *Organometallics*, **4**, 2228–2230.