$wR = 0.033$
$S = 1.79$
2166 reflections
165 parameters
$w = 1/\sigma^2(|F_o|)$
$(\Delta/\sigma)_{\max} = 0.095$

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	$U_{\rm eq}$
Ni	0.16989 (3)	0.00730 (2)	0.03877 (3)	0.0234 (1)
P(1)	0.09649 (7)	0.08798 (5)	0.22127 (6)	0.0243 (2)
P(2)	0.23627 (7)	−0.07689 (5)	−0.14487 (6)	0.0232 (2)
N(1)	0.3709 (3)	0.2106 (2)	−0.0081 (3)	0.0468 (8)
N(2)	0.0258 (3)	−0.2143 (2)	0.1149 (3)	0.0497 (9)
C(1)	0.2938 (3)	0.1338 (2)	0.0077 (2)	0.0318 (8)
C(2)	0.0698 (3)	−0.1313 (2)	0.0854 (2)	0.0286 (7)
C(3)	−0.0926 (3)	0.0564 (2)	0.2671 (2)	0.0275 (7)
C(4)	0.1952 (4)	0.0359 (3)	0.3640 (3)	0.0430 (10)
C(5)	0.1115 (4)	0.2448 (2)	0.2310 (3)	0.0382 (9)
C(6)	0.3955 (3)	−0.0233 (3)	−0.2304 (3)	0.0371 (9)
C(7)	0.2725 (3)	−0.2321 (2)	−0.1398 (3)	0.0351 (9)

Table 2. *Selected geometric parameters* (Å, °)

Ni—P(1)	2.190 (1)	Ni—P(2)	2.198 (1)
Ni—C(1)	1.869 (3)	Ni—C(2)	1.896 (3)
P(1)—C(3)	1.830 (3)	P(1)—C(4)	1.813 (4)
P(1)—C(5)	1.806 (3)	P(2)—C(3′)	1.824 (3)
P(2)—C(6)	1.810 (3)	P(2)—C(7)	1.810 (3)
N(1)—C(1)	1.140 (4)	N(2)—C(2)	1.076 (4)
P(1)—Ni—P(2)	178.0 (1)	P(1)—Ni—C(1)	90.3 (1)
P(1)—Ni—C(2)	89.4 (1)	P(2)—Ni—C(1)	91.4 (1)
P(2)—Ni—C(2)	89.0 (1)	C(1)—Ni—C(2)	170.8 (2)
Ni—P(1)—C(3)	115.4 (1)	Ni—P(1)—C(4)	113.1 (2)
Ni—P(1)—C(5)	116.3 (2)	C(3)—P(1)—C(4)	101.4 (2)
C(3)—P(1)—C(5)	104.7 (2)	C(4)—P(1)—C(5)	104.2 (2)
Ni—P(2)—C(3′)	109.1 (1)	Ni—P(2)—C(6)	119.3 (2)
Ni—P(2)—C(7)	117.3 (1)	C(3′)—P(2)—C(6)	101.8 (2)
C(3′)—P(2)—C(7)	106.1 (2)	C(6)—P(2)—C(7)	101.5 (2)
Ni—C(1)—N(1)	178.2 (3)	Ni—C(2)—N(2)	173.1 (3)
P(1)—C(3)—P(2′)	118.6 (2)		

The structure was solved by Patterson and Fourier methods and refined by full-matrix least squares. The non-H atoms were assigned anisotropic displacement parameters. H atoms were located in a low-angle $\Delta\rho$ map and their coordinates and isotropic displacement parameters were refined. All calculations were performed using the *GX* program package (Mallinson & Muir, 1985).

We thank Dr H. A. Mirza and Professor R. J. Puddephatt for a gift of crystals and the SERC for an equipment grant.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1128). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 1535–1537

(2-Acetylbicyclo[2.2.1]heptyl-C^3,O)(2,2′-bipyridyl)palladium(II) Trifluoromethanesulfonate 0.5-Diethyl Ether Solvate

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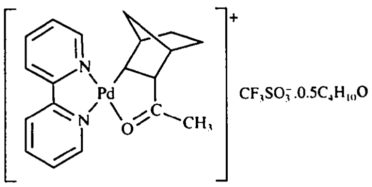
(*Received* 25 *March* 1994; *accepted* 20 *April* 1994)

Abstract

The norbornyl moiety in the title compound, [Pd(C_9H_{13}-O)($C_{10}H_8N_2$)]CF_3SO_3.0.5$C_4H_{10}O$, is selectively *cis*, *exo* substituted on the 2 and 3 positions with the acetyl substituent bonded intramolecularly *via* its O atom to the palladium(II) centre.

Comment

The title compound (I) was prepared *via* norbornene insertion into the Pd—C(acetyl) bond. In most cases, such alkene-insertion products are unstable and readily undergo β-hydrogen elimination (Dekker, Elsevier, Vrieze, van Leeuwen & Roobeek, 1992). The structure determination was undertaken to establish the bonding of the 3-acetyl-2-norbornyl group and the stereochemistry of the insertion reaction.



(I)

The unit cell contains four cations of the Pd complex (Fig. 1), four trifluoromethanesulfonate anions and two diethyl ether molecules disordered over a crystallographic inversion centre. The cation is a 2,3-*cis*,*exo*-substituted norbornyl moiety with a palladium(II) centre and an acetyl substituent. The carbonyl group is coordinated to the metal [Pd—O 2.028 (3) Å] resulting in a five-membered Pd—C—C—C—O chelate ring. The geometry about the Pd atom is square planar, comprising the 2,2′-bipyridyl ligand which occupies two adjacent positions [Pd—N1 and Pd—N2 = 2.014 (4) and 2.100 (4) Å, respectively; N1—Pd—N2 = 79.60 (14)°] and the *C*,*O* chelate-bonded ligand. The Pd—C11 distance for the latter ligand [2.019 (3) Å] is comparable to those found for other sp^3 C atoms *trans* to an sp^2 N atom [2.020 (5) Å; Markies *et al.*, 1991]. The C18—O bond distance [1.235 (5) Å] is typical for a carbonyl group and is comparable with the C—O distance in the analogous norbornyl-inserted product $[Pd(C_7H_{10}COMe)(PPh_3)_2]BF_4$ [1.240 (10) Å; Brumbaugh, Whittle, Parvez & Sen, 1990] and the dicyclopentadiene-inserted product $[Pd(C_{10}H_{12}COMe)$-(2,2′-bipyridyl)]CF_3SO_3 [1.249 (6) Å; Markies, Rietveld, Boersma, Spek & van Koten, 1992].

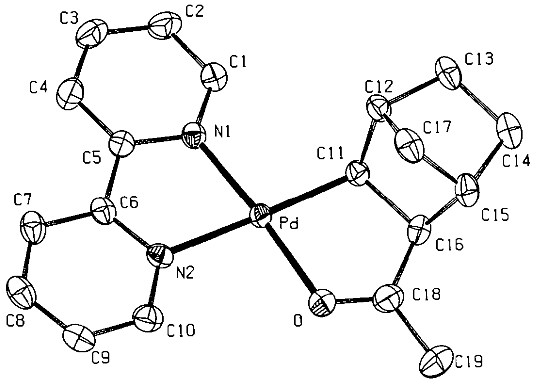


Fig. 1. View of the $[Pd(C_9H_{13}O)(C_{10}H_8N_2)]$ cation with displacement ellipsoids drawn at the 50% probability level. H atoms, the anion and the disordered diethyl ether have been omitted.

Experimental

The preparation of the title compound has been described by Markies, Rietveld, Boersma, Spek & van Koten (1992). Crystals were obtained from a 1:1 diethyl ether/methanol solution.

Crystal data

$[Pd(C_9H_{13}O)(C_{10}H_8N_2)]$-
$CF_3SO_3.0.5C_4H_{10}O$
$M_r = 585.94$
Monoclinic
$P2_1/c$
$a = 14.3658$ (12) Å
$b = 11.0537$ (6) Å
$c = 19.3767$ (7) Å
$\beta = 128.707$ (5)°
$V = 2401.1$ (3) Å^3
$Z = 4$
$D_x = 1.621$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11$–14°
$\mu = 0.90$ mm^{-1}
$T = 150$ K
Block
0.50 × 0.50 × 0.25 mm
Yellowish

Data collection

Enraf–Nonius Turbo CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.76$, $T_{max} = 1.00$
6075 measured reflections
5168 independent reflections

4095 observed reflections [$I > 2.5\sigma(I)$]
$R_{int} = 0.027$
$\theta_{max} = 27.5°$
$h = -18 \rightarrow 18$
$k = 0 \rightarrow 14$
$l = -18 \rightarrow 25$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
$R = 0.037$
$wR = 0.043$
$S = 1.40$
4095 reflections
328 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{max} = 0.500$
$\Delta\rho_{max} = 0.830$ e Å^{-3}
$\Delta\rho_{min} = -0.590$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from Cromer & Mann (1968)

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

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

	x	y	z	U_{eq}
Pd	0.04125 (2)	0.10808 (2)	0.12473 (2)	0.0198 (1)
O	0.2177 (2)	0.1285 (2)	0.22633 (18)	0.0328 (9)
N1	−0.1303 (3)	0.0789 (3)	0.0193 (2)	0.0211 (9)
N2	0.0458 (3)	−0.0785 (3)	0.1067 (2)	0.0231 (9)
C1	−0.2147 (3)	0.1647 (3)	−0.0259 (3)	0.0281 (11)
C2	−0.3292 (3)	0.1398 (4)	−0.1012 (3)	0.0377 (14)
C3	−0.3590 (4)	0.0220 (4)	−0.1309 (3)	0.0388 (14)
C4	−0.2730 (3)	−0.0675 (4)	−0.0847 (3)	0.0326 (12)
C5	−0.1594 (3)	−0.0377 (3)	−0.0103 (2)	0.0227 (11)
C6	−0.0608 (3)	−0.1256 (3)	0.0395 (2)	0.0223 (11)
C7	−0.0748 (4)	−0.2481 (3)	0.0200 (3)	0.0292 (11)
C8	0.0236 (4)	−0.3232 (4)	0.0706 (3)	0.0311 (12)
C9	0.1320 (4)	−0.2752 (3)	0.1385 (3)	0.0318 (12)
C10	0.1406 (3)	−0.1528 (3)	0.1556 (3)	0.0291 (11)
C11	0.0316 (3)	0.2851 (3)	0.1454 (2)	0.0220 (10)
C12	−0.0355 (3)	0.3050 (3)	0.1813 (3)	0.0248 (11)
C13	−0.0513 (4)	0.4423 (3)	0.1848 (3)	0.0307 (11)
C14	0.0768 (4)	0.4863 (3)	0.2617 (3)	0.0332 (14)
C15	0.1492 (4)	0.3691 (3)	0.2937 (3)	0.0297 (12)
C16	0.1602 (3)	0.3309 (3)	0.2222 (2)	0.0248 (11)
C17	0.0593 (4)	0.2760 (3)	0.2788 (3)	0.0317 (14)
C18	0.2500 (3)	0.2320 (4)	0.2564 (3)	0.0320 (12)
C19	0.3795 (4)	0.2556 (5)	0.3280 (3)	0.0528 (16)
O5	1/2	1/2	1/2	0.116 (4)
C21	0.4076 (12)	0.5792 (13)	0.4538 (10)	0.075 (5)

C22	0.382 (3)	0.652 (4)	0.391 (3)	0.27 (2)
C23	0.608 (3)	0.336 (2)	0.593 (3)	0.163 (12)
C24	0.5243 (17)	0.4139 (12)	0.5576 (11)	0.081 (6)
S1	0.32227 (10)	0.48444 (9)	0.11744 (8)	0.0399 (3)
F1	0.3195 (4)	0.3775 (3)	−0.0037 (3)	0.0906 (17)
F2	0.3594 (4)	0.5691 (4)	0.0110 (3)	0.1014 (17)
F3	0.4919 (3)	0.4434 (4)	0.1082 (3)	0.114 (2)
O2	0.3415 (3)	0.3673 (3)	0.1569 (2)	0.0579 (11)
O3	0.1994 (3)	0.5149 (3)	0.0504 (2)	0.0455 (10)
O4	0.3945 (3)	0.5796 (3)	0.1782 (2)	0.0656 (11)
C20	0.3771 (5)	0.4669 (6)	0.0550 (5)	0.073 (3)

Table 2. *Selected geometric parameters* (Å, °)

Pd—O	2.028 (3)	C16—C18	1.493 (6)
Pd—N1	2.014 (4)	C18—C19	1.492 (8)
Pd—N2	2.100 (3)	O5—C21	1.358 (17)
Pd—C11	2.019 (3)	O5—C24	1.336 (16)
O—C18	1.235 (5)	S1—O2	1.440 (3)
N1—C1	1.345 (6)	S1—O3	1.431 (4)
N1—C5	1.366 (5)	S1—O4	1.429 (3)
N2—C6	1.348 (5)	S1—C20	1.820 (9)
N2—C10	1.347 (6)	F1—C20	1.333 (8)
C5—C6	1.474 (5)	F2—C20	1.340 (8)
C11—C16	1.564 (5)	F3—C20	1.313 (10)
O—Pd—N1	175.41 (14)	C6—N2—C10	118.8 (3)
O—Pd—N2	96.94 (13)	N1—C1—C2	122.7 (3)
O—Pd—C11	84.19 (13)	N1—C5—C4	121.0 (4)
N1—Pd—N2	79.60 (14)	N1—C5—C6	115.5 (3)
N1—Pd—C11	99.45 (15)	N2—C6—C5	115.1 (3)
N2—Pd—C11	176.33 (18)	N2—C6—C7	121.6 (4)
Pd—O—C18	115.7 (3)	N2—C10—C9	122.0 (4)
Pd—N1—C1	125.5 (3)	Pd—C11—C12	111.9 (3)
Pd—N1—C5	115.9 (3)	Pd—C11—C16	108.4 (3)
C1—N1—C5	118.5 (4)	O—C18—C16	120.2 (4)
Pd—N2—C6	113.9 (3)	O—C18—C19	118.9 (4)
Pd—N2—C10	127.2 (3)		

X-ray data were collected for a fragment cut from a large crystal using a scan width $\Delta\omega$ of $(0.99 + 0.35\tan\theta)^\circ$. The transmission range (0.76–1.00) of the absorption correction is comparable with the intensity variations observed in three 360° ψ scans for close-to-axial reflections (0.80–1.00). The diethyl ether molecule was refined with a disorder model. H atoms were refined riding on their carrier atoms with three common isotropic atomic displacement parameters.

Data collection: locally modified CAD-4 (Version 5) software. Cell refinement: *SET*4 (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1993). Program(s) used to solve structure: *DIRDIF*92 (Beurskens *et al.*, 1992). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *PLATON* (Spek, 1990).

This work was supported in part by the Netherlands Foundation for Chemical Research (SON) with financial aid from the Netherlands Organization for Scientific Research (NWO)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1183). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 1537–1540

trans-Bis(3-hydroxy-2-phenyl-4*H*-1-benzopyran-4-onato)bis(pyridine)nickel(II)

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(*Received* 20 *September* 1994; *accepted* 17 *January* 1995)

Abstract

3-Hydroxyflavone and bipyridyl ligands coordinate to the Ni^{II} centre in an octahedral arrangement, giving the title complex, [Ni($C_{15}H_9O_3$)$_2$(C_5H_5N)$_2$]. Since the Ni atom lies on a centre of symmetry, all the *trans* O—Ni—O and N—Ni—N bond angles are 180°; those of O—Ni—N are close to 90°. The Ni—O(carbonyl), Ni—O(hydroxy) and Ni—N distances are 2.067 (2), 2.023 (2) and 2.180 (2) Å, respectively. All the C—C bond distances are normal with values between 1.366 (3) and 1.406 (4) Å, except those which are close to the coordinated C—O bonds: C(1)—C(2), C(2)—C(3) and C(9)—C(10) are 1.457 (4), 1.445 (3) and 1.462 (4) Å, respectively. All rings are almost planar, except those involving coordinated O atoms. The dihedral angles between the flavonato planes and the pyridyl ligands are