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

Ni—O1	2.046 (2)	O4^{v}—H1	0.93 (3)
Ni—O4	2.074 (2)	O5^{i}—H3^{i}	0.91 (5)
Ni—O5	2.094 (3)	O4^{v}—H2^{i}	0.84 (5)
C—O1	1.292 (5)	O5^{i}—H4^{i}	1.02 (3)
C—O2	1.275 (4)	O3^{ii}···H1^{vii}	1.65 (4)
C—O3	1.289 (4)	O2^{iii}···H2^{iii}	1.85 (5)
K^{i}···O1^{i}	2.768 (3)	O2^{i}···O4^{v}	2.678 (4)
K^{i}···O3^{ii}	2.723 (3)	O3^{v}···O4^{v}	2.565 (4)
K^{i}···O5^{i}	2.978 (3)	O3^{ii}···H4^{viii}	1.67 (3)
K^{i}···O1	2.779 (3)	O2^{iii}···H3^{vii}	1.84 (5)
K^{i}···O2^{iii}	2.699 (3)	O2^{ix}···O5^{i}	2.727 (3)
K^{i}···O4^{iv}	3.094 (3)	O3^{iv}···O5^{i}	2.658 (4)
K^{i}···O4^{v}	2.814 (3)		
O1—Ni—O4^{v}	89.0 (1)	O5^{i}···K^{i}···O2^{iii}	80.0 (1)
O1—Ni—O5	88.7 (1)	O5^{i}···K^{i}···O4^{v}	128.2 (1)
O4^{v}—Ni—O5	89.5 (1)	O1···K^{i}···O2^{iii}	126.9 (1)
O1—C—O2	119.5 (3)	O1···K^{i}···O4^{iv}	66.6 (1)
O1—C—O3	120.5 (3)	O5^{i}···K^{i}···O4^{iv}	86.1 (1)
O2—C—O3	120.0 (3)	O1^{i}···K^{i}···O4^{v}	70.9 (1)
O1^{i}···K^{i}···O3^{ii}	148.9 (1)	O1···K^{i}···O4^{v}	62.2 (1)
O1^{i}···K^{i}···O5^{i}	60.4 (1)	O2^{iii}···K^{i}···O4^{iv}	164.9 (1)
O1^{i}···K^{i}···O1	112.2 (1)	O2^{iii}···K^{i}···O4^{v}	79.6 (1)
O1^{i}···K^{i}···O2^{iii}	85.2 (1)	O4^{iv}···K^{i}···O4^{v}	104.9 (1)
O1^{i}···K^{i}···O4^{iv}	82.7 (1)	H1—O4^{v}—H2^{i}	100 (5)
O3^{ii}···K^{i}···O5^{i}	88.7 (1)	O4^{iv}—H1^{vi}···O3^{iv}	165 (4)
O3^{ii}···K^{i}···O1	96.8 (1)	O4^{iv}—H2···O2	168 (6)
O3^{ii}···K^{i}···O2^{iii}	86.1 (1)	H4^{i}—O5^{i}—H3^{i}	99 (4)
O3^{ii}···K^{i}···O4^{iv}	99.6 (1)	O5^{vii}—H3^{vii}···O2^{iii}	164 (4)
O3^{ii}···K^{i}···O4^{v}	136.3 (1)	O5^{i}—H4^{i}···O3^{iv}	165 (3)
O5^{i}···K^{i}···O1	152.7 (1)		

Symmetry codes: (i) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iii) $x, 1+y, z$; (iv) $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{1}{2}$; (v) $1-x, 1-y, 1-z$; (vi) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (vii) $x-\frac{1}{2}, \frac{3}{2}-y, \frac{1}{2}+z$; (viii) $-\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (ix) $-x, 1-y, 1-z$.

All non-H atoms were found in a Patterson map. The H atoms were located using difference Fourier syntheses. Refinement of all positional and displacement parameters (isotropic for H atoms and anisotropic for non-H atoms) was carried out using a full-matrix least-squares method. Data reduction including Lorentz–polarization correction: *DATNEU* (Fröhlich & Tebbe, 1979). Program used for empirical absorption correction: *SDP* (Enraf–Nonius, 1985). Program used to solve the structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *SCHAKAL* (Keller, 1988).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SH1076). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Rhodium(I) Complexes with 1,5-Diazabicyclo[4.3.0]non-5-ene (dbn) as a Ligand: [RhCl(nbd)(dbn)] (I) and [Rh(dbn)$_2$(cod)][PF$_6$] (II) (nbd = Norbornadiene, cod = 1,5-Cyclooctadiene)

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Abstract

Both of the complexes studied, (η^4-bicyclo[2.2.1]hepta-2,5-diene)chloro(1,5-diazabicyclo[4.3.0]non-5-ene-N^5)-rhodium(I), [RhCl(C_7H_8)($C_7H_{12}N_2$)], (I), and (η^4-1,5-cyclooctadiene)bis(1,5-diazabicyclo[4.3.0]non-5-ene-N^5)rhodium hexafluorophosphate, [Rh($C_7H_{12}N_2$)-(C_8H_{12})][PF$_6$], (II), show planar quadratically coordinated Rh atoms with dbn as and N-donor ligand. Two different conformations of the dbn ligand are present in each complex. The Rh–ligand bond distances and angles depend on the different N, Cl and diene coordination of the metal atoms.

Comment

In a recent paper we reported [RhCl(cod)(dbn)], the first Rh^{I} complex with dbn as a ligand to be characterized by means of an X-ray analysis (Flörke, Ortmann & Haupt, 1992). This and related complexes are used successfully as catalysts for the polymerization of phenylacetylene. Molecular weights up to 900 000 with 86% turnover have been achieved (Kowalzik, 1993). In the course of these investigations, the new compounds [RhCl(nbd)(dnb)], (I), and [Rh(dbn)$_2$(cod)][PF$_6$], (II), were crystallized.

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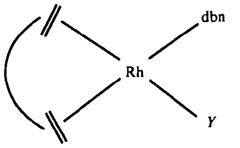

(I); Y = Cl
(II); Y = dbn

In both of these complexes, the central Rh atom is fourfold coordinated: by an N atom from the dbn ligand, the terminal Cl atom, and the two nbd double bonds [C(11)=C(16) and C(13)=C(14)] in (I), and by two N atoms from the two dbn ligands and the two cod double bonds [C(9)=C(10)] in (II).

The Rh atom in (I) lies 0.014 Å above the best plane defined by the four ligating groups Cl(1), N(1), X(1) and X(2), where X(1) and X(2) denote the midpoints of the C(11)=C(16) and C(13)=C(14) bonds, respectively. The Rh–ligand bond distances [Rh(1)—Cl(1) 2.375 (2), Rh(1)—N(1) 2.108 (4), Rh(1)—X(1) 2.006 (5) and Rh—X(2) 1.981 (5) Å] are the same as for [RhCl(cod)(dbn)] within experimental error [2.375 (1), 2.103 (4), 2.001 (5) and 1.978 (5) Å, respectively (Flörke, Ortmann & Haupt, 1992)]. The shorter Rh—X bond is, in both cases, *trans* to the Rh—Cl bond, as the Cl atom is a weak π donor. However, the bond angles at the Rh atom change considerably on going from the cod to the nbd complex. As the limiting chelate angle of the nbd ligand is smaller than that of the cod ligand, the X(1)—Rh1—X(2) angle is reduced from 88.0 (2)° in the cod complex to 72.0 (2)° in (I). The Cl—Rh—N angle opposite increases from 87.7 (1)° in the cod complex to 95.0 (1)° in (I), while the remaining X—Rh—N and X—Rh—Cl angles in (I) are 97.1 (2) and 96.2 (2)°, respectively. The sum of the four plane angles at the Rh atom is 360° in both complexes. Similar geometries are known for other transition metal–nbd complexes, *e.g.* [Rh(nbd)(AcO)]$_2$ (Reis, Willi, Siegel & Tani, 1979) and [W(CO)$_4$(nbd)] (Grevels, Jacke, Betz, Krüger & Tsay, 1989). The mean value of 1.380 Å for the nbd double bonds is in accordance with the elongation of transition-metal-coordinated olefinic bonds (Itho, Oshima, Jameson, Lewis & Ibers, 1981). The C(2) position of the dbn ligand is disordered over two sites, C(21) and C(22), with site occupation factors of 0.5 each. The same effect was observed for [RhCl(cod)(dbn)], and results from two different conformations of the ligand. The anisotropic displacement parameters of C(1) indicate a similar disorder, but in this case the two positions could not be resolved.

The cation of (II) lies on a crystallographic twofold axis which runs through the Rh atom and the centre of the cod ligand; hence the Rh atom lies exactly in the best plane through the four ligands. For this nearly undistorted planar quadratic ligand arrangement, the angles at the central atom are X—Rh(1)—N(1) 91.1 (2), N(1)—Rh(1)—N(1$^{\rm i}$) 90.4 (1) and $X^{\rm i}$—Rh—X 87.6 (2)°, where X represents the midpoint of the C(9)=C(10) double bond [symmetry code: (i) $-x, y, \frac{1}{2}-z$]. The last of these angles shows the greatest deviation from the ideal value of 90° and agrees well with observations for other Rh(cod) complexes, *e.g.* 87.8 (2) (Halesha, Reddy, Sudhakar Rao & Manohar, 1983), 86.8 (5) (Bonnaire, Manoli, Potvin, Platzer, Goasdoue & Davoust, 1982) and 88.0 (2)° for the complex [RhCl(cod)(dbn)] mentioned above (Flörke, Ortmann & Haupt, 1992). The cod ligand has the expected skewed conformation which is known for other metal–cod complexes (Halesha, Reddy, Sudhakar Rao & Manohar, 1983, and references therein). The C(2) position of the dbn ligand of (II) is also disordered. Here the two sites, C(21) and C(22), have site occupation factors of 0.73 and 0.27, respectively. The Rh–ligand distances are 2.019 (3) for Rh(1)—X and 2.129 (2) Å for Rh(1)—N(1). Compared to the analogous distances for [RhCl(cod)(dbn)] [2.001 (5) and 2.103 (4) Å, respectively], the Rh—X bond tends to be longer and the Rh—N bond is significantly increased by introduction of a

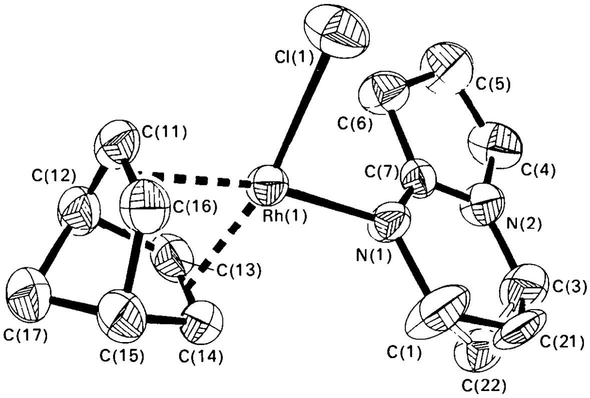

Fig. 1. Molecular structure of the complex [RhCl(nbd)(dbn)] (I). Displacement ellipsoids are plotted at the 50% probability level.

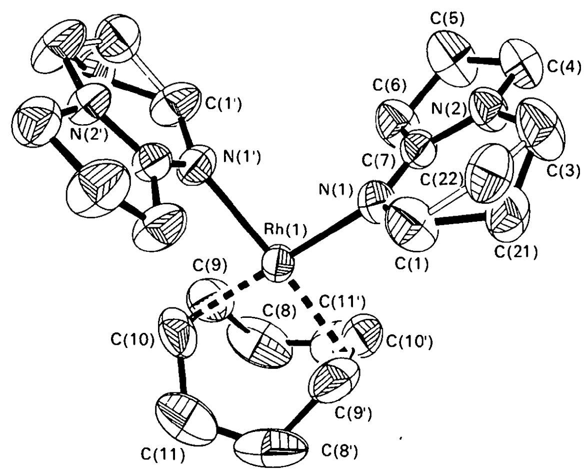

Fig. 2. Molecular structure of the cation [Rh(dbn)$_2$(cod)] (II). Displacement ellipsoids are plotted at the 50% probability level. [Symmetry code: (i) $-x, y, \frac{1}{2}-z$.]

second dbn ligand instead of Cl. The hexafluorophosphate anion is situated on an inversion centre and is a nearly undistorted PF_6^- octahedron.

Experimental

Compound (I) was prepared by the reaction of [Rh(nbd)Cl]$_2$ with dbn in tetrahydrofuran solution at 298 K. Compound (II) was prepared by the reaction of dbn and Rh(cod)Cl$_2$ with addition of $TlPF_6$ in CH_2Cl_2 solution at 298 K.

Compound (I)

Crystal data

[RhCl($C_7H_{12}N_2$)(C_7H_8)]
$M_r = 354.68$
Monoclinic
$P2_1/c$
$a = 14.139$ (4) Å
$b = 7.378$ (2) Å
$c = 14.855$ (4) Å
$\beta = 114.22$ (2)°
$V = 1413.2$ (7) Å^3
$Z = 4$
$D_x = 1.667$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8$–16°
$\mu = 1.381$ mm^{-1}
$T = 293$ (2) K
Plate
0.30 × 0.16 × 0.05 mm
Yellow

Data collection

Siemens $R3m/V$ diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scans
$T_{\min} = 0.834$, $T_{\max} = 0.977$
3411 measured reflections
3282 independent reflections
2166 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0232$
$\theta_{\max} = 27.55°$
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 9$
$l = -19 \rightarrow 17$
3 standard reflections monitored every 400 reflections
intensity variation: none

Refinement

Refinement on F^2
$R(F) = 0.0482$
$wR(F^2) = 0.0909$
$S = 1.074$
3282 reflections
172 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.4404P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 0.734$ e Å^{-3}
$\Delta\rho_{\min} = -0.559$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
Rh(1)	0.19649 (3)	0.16119 (6)	0.00860 (3)	0.03668 (14)
Cl(1)	0.29829 (14)	−0.1003 (2)	0.01866 (13)	0.0635 (5)
N(1)	0.2989 (4)	0.2692 (6)	0.1453 (3)	0.0441 (12)
N(2)	0.3802 (4)	0.2771 (7)	0.3192 (3)	0.0491 (13)
C(1)	0.3466 (7)	0.4466 (10)	0.1464 (5)	0.085 (3)
C(21)†	0.4247 (13)	0.5119 (23)	0.2384 (16)	0.065 (5)
C(22)†	0.3731 (13)	0.5528 (23)	0.2297 (11)	0.049 (4)
C(3)	0.4204 (6)	0.4602 (9)	0.3292 (5)	0.065 (2)
C(4)	0.3902 (6)	0.1656 (9)	0.4026 (4)	0.064 (2)
C(5)	0.3439 (6)	−0.0118 (10)	0.3590 (5)	0.071 (2)
C(6)	0.2824 (5)	0.0216 (8)	0.2486 (4)	0.052 (2)
C(7)	0.3204 (4)	0.2000 (7)	0.2309 (4)	0.0364 (12)
C(11)	0.0609 (4)	0.0537 (8)	−0.1046 (4)	0.0446 (14)
C(12)	−0.0160 (5)	0.1760 (8)	−0.0843 (4)	0.0502 (15)
C(13)	0.0615 (4)	0.2924 (8)	−0.0009 (4)	0.049 (2)
C(14)	0.1118 (4)	0.4024 (8)	−0.0430 (4)	0.0465 (14)
C(15)	0.0664 (5)	0.3526 (9)	−0.1527 (4)	0.055 (2)
C(16)	0.1109 (5)	0.1615 (9)	−0.1469 (4)	0.0481 (14)
C(17)	−0.0468 (5)	0.3094 (9)	−0.1712 (4)	0.058 (2)

† Site occupancy = 0.5.

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

X(1) and X(2) denote the midpoints of the C(11)═C(16) and C(13)═C(14) bonds, respectively.

Rh(1)—C(13)	2.092 (6)	Rh(1)—Cl(1)	2.375 (2)
Rh(1)—C(14)	2.106 (6)	Rh(1)—X(1)	2.006 (5)
Rh(1)—N(1)	2.108 (4)	Rh(1)—X(2)	1.981 (5)
Rh(1)—C(11)	2.118 (5)	C(11)—C(16)	1.375 (8)
Rh(1)—C(16)	2.123 (5)	C(13)—C(14)	1.386 (8)
N(1)—Rh(1)—Cl(1)	95.0 (1)	X(1)—Rh(1)—Cl(1)	96.2 (2)
X(1)—Rh(1)—X(2)	72.0 (2)	X(2)—Rh(1)—N(1)	97.1 (2)

Compound (II)

Crystal data

[Rh($C_7H_{12}N_2$)$_2$(C_8H_{12})][PF_6]
$M_r = 604.43$
Monoclinic
$C2/c$
$a = 18.245$ (3) Å
$b = 8.3330$ (10) Å
$c = 16.535$ (3) Å
$\beta = 94.920$ (10)°
$V = 2504.6$ (7) Å^3
$Z = 4$
$D_x = 1.603$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 28 reflections
$\theta = 7.5$–20°
$\mu = 0.809$ mm^{-1}
$T = 293$ (2) K
Prism
0.42 × 0.40 × 0.20 mm
Yellow

Data collection

Siemens $R3m/V$ diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scans
$T_{\min} = 0.398$, $T_{\max} = 0.451$
5756 measured reflections
2883 independent reflections
2495 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0254$
$\theta_{\max} = 27.56°$
$h = -23 \rightarrow 23$
$k = -10 \rightarrow 10$
$l = 0 \rightarrow 21$
3 standard reflections monitored every 400 reflections
intensity variation: none

Refinement

Refinement on F^2
$R(F) = 0.0347$
$wR(F^2) = 0.0904$
$S = 1.046$
2883 reflections
166 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 2.3655P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 0.465$ e Å^{-3}
$\Delta\rho_{\min} = -0.380$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (II)

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

	x	y	z	$U_{\rm eq}$
Rh(1)	0	0.13238 (3)	1/4	0.04222 (12)
N(1)	−0.08157 (12)	−0.0477 (3)	0.2250 (2)	0.0477 (5)
N(2)	−0.14580 (13)	−0.2118 (3)	0.12792 (15)	0.0490 (6)
C(1)	−0.1343 (2)	−0.0959 (5)	0.2825 (2)	0.0653 (9)
C(21)†	−0.2066 (4)	−0.1394 (17)	0.2445 (5)	0.066 (2)
C(22)‡	−0.1882 (12)	−0.2208 (39)	0.2586 (11)	0.065 (5)
C(3)	−0.2060 (2)	−0.2520 (5)	0.1756 (2)	0.0761 (12)
C(4)	−0.1481 (2)	−0.2445 (5)	0.0414 (2)	0.0623 (8)
C(5)	−0.0744 (2)	−0.1894 (6)	0.0190 (2)	0.0780 (11)
C(6)	−0.0473 (2)	−0.0721 (5)	0.0834 (2)	0.0633 (9)
C(7)	−0.09274 (15)	−0.1087 (3)	0.1536 (2)	0.0448 (6)
C(8)	0.0417 (3)	0.4474 (5)	0.1726 (3)	0.099 (2)
C(9)	0.0783 (2)	0.3128 (5)	0.2255 (3)	0.0710 (10)
C(10)	0.0746 (2)	0.3018 (4)	0.3070 (3)	0.0709 (10)
C(11)	0.0355 (3)	0.4152 (6)	0.3574 (3)	0.0931 (14)
P(1)	1/4	1/4	1/2	0.0619 (3)
F(1)	0.3331 (2)	0.2424 (6)	0.4860 (2)	0.1489 (15)
F(2)	0.2606 (2)	0.1064 (3)	0.5619 (2)	0.1218 (12)
F(3)	0.2330 (2)	0.1307 (3)	0.4275 (2)	0.1227 (13)

† Site occupancy 0.73 (3).
‡ Site occupancy 0.27 (3).

Table 4. *Selected geometric parameters* (Å, °) *for* (II)

X represents the midpoint of the C(9)═C(10) double bond.

Rh(1)—C(10)	2.124 (3)	Rh(1)—X	2.019 (3)
Rh(1)—N(1)	2.129 (2)	C(9)—C(10)	1.359 (6)
Rh(1)—C(9)	2.136 (3)		
N(1)—Rh(1)—N(1$^{\rm i}$)	90.4 (1)	$X^{\rm i}$—Rh(1)—X	87.6 (2)
N(1)—Rh(1)—X	91.1 (2)		

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

Program used to solve structures: *SHELXS*86 (Sheldrick, 1990). Program used to refine structures: *SHELXL*93 (Sheldrick, 1994).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1015). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(dimethylglyoximato-*N*,*N*′)(triphenylphosphine-*P*)vinylrhodium(III)

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Abstract

The title structure {bis[2,3-butanedial dioximato-(1 −)-*N*,*N*′](triphenylphosphine-*P*)vinylrhodium(III), [$Rh(C_2H_3)(C_4H_7N_2O_2)_2(C_{18}H_{15}P)$]} consists of discrete complexes in which the Rh atom displays distorted octahedral coordination, where the two dimethylglyoxime ligands lie in the equatorial plane and the vinyl and triphenylphosphine groups occupy the axial positions. The Rh atom is displaced by 0.123 (1) Å from the mean plane through the four oxime N donors. The average Rh—N distance is 1.957 (2) Å, while the axial Rh—P and Rh—C distances are 2.447 (1) and 2.035 (3) Å, respectively. Comparison of the results with those obtained previously for the analogous ethyl compound indicates a lower *trans* influence of the vinyl ligand in the axial P—Rh—C fragment.

Comment

This work is part of a more general study aimed at understanding the mutual influence of ligands in organometallic compounds (Steinborn, 1992). The *trans* influence of organic ligands is of particular interest. Recent NMR investigations of complexes [$Rh(dmgH)_2(PPh_3)R$] (dmgH is the monoanion of dimethylglyoxime, R is an organo group) (Steinborn & Ludwig, 1993) demonstrates that the *trans* influence of the organo group R, as measured by $^1J(^{103}Rh-^{31}P)$ coupling constants, is nearly the same for the sp^3-hybridized ethyl group and the sp^2-

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