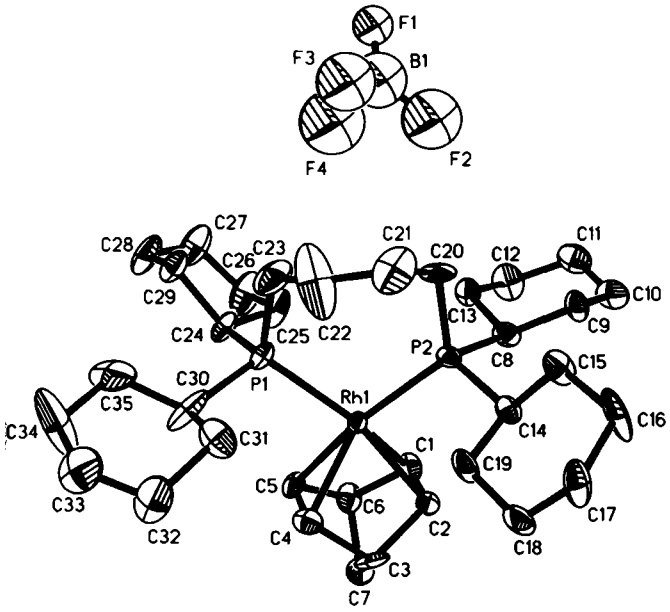
Crystal structure of 1,4-bis(dicyclohexylphosphino)-butane rhodium(I)-norborna-2,5-diene tetrafluoroborate, $C_{35}H_{60}BF_4P_2Rh$, and of 1,4-bis(dicyclohexylphosphino)-butane rhodium(I)-(Z,Z)-cycloocta-1,5-diene tetrafluoroborate, $C_{36}H_{64}BF_4P_2Rh$

R. Kempe*, A. Spannenberg and D. Heller

Institut für Organische Katalyseforschung an der Universität Rostock e.V., Buchbinderstr. 5-6, D-18055 Rostock, Germany

Received August 21, 2000, CCDC-No. 1267/515 and CCDC-No. 1267/516



Abstract

$C_{35}H_{60}BF_4P_2Rh$, orthorhombic, $Pna2_1$ (No. 33), a = 19.225(4) Å, b = 10.487(2) Å, c = 17.636(4) Å, V = 3555.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.054, $wR_{all}(F^2)$ = 0.119, T = 293 K.

$C_{36}H_{64}BF_4P_2Rh$, monoclinic, $P12_1/c1$ (No. 14), a = 19.737(4) Å, b = 10.058(2) Å, c = 20.706(4) Å, β = 115.86(3)°, V = 3698.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.044, $wR_{all}(F^2)$ = 0.117, T = 200 K.

Source of material

Standard procedure according to [1] – the bisphosphine ligand is commercially available.

Discussion

Unexpected differences between the title compounds in the catalytic hydrogenation of the diolefines norborna-2,5-diene and (Z,Z)-cycloocta-1,5-diene (compare with [2]) motivated us to determine the crystal structures. It is well known that the double bonds of the diolefines are not coordinated perpendicular to the P,Rh,P plane. The structure of the cod complex with the 1,4-bis(diphenylphosphino)-butane shows an dihedral angle of 7.7° between the planes P,Rh,P and X,Rh,X (X = centroid of the double bond) [3]. In the case of the 1,4-bis(dicyclohexylphosphino)-butane we found for the dihedral angle of the cod-complex 12.5° and for the nbd-complex 17.0°. Thereby it had to be considered, that the substitution of the phenyl groups at the phosphorus by the cyclohexyl groups leads to an increasing sterical demand and to an increasing of the electron density at the rodium.

1. 1,4-bis(dicyclohexylphosphino)-butane rhodium(I)-norborna-2,5-diene tetrafluoroborate, $C_{35}H_{60}BF_4P_2Rh$

Table 1. Data collection and handling.

Crystal:	red prism, size 0.1 × 0.1 × 0.1 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.15 cm^{-1}
Diffractometer, scan mode:	STOE IPDS, 100 exposures, Δφ = 2°
$2\theta_{max}$:	48.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12236, 5574
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2797
$N(param)_{refined}$:	363
Programs:	SHELXS-86 [4], SHELXL-93 [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4a	0.7750(6)	0.007(2)	0.038(1)	0.070
H(2)	4a	0.8090(5)	0.232(1)	0.068(1)	0.054
H(3)	4a	0.8450(6)	0.234(1)	0.211(1)	0.070
H(4)	4a	0.7189(5)	0.205(1)	0.248(1)	0.060
H(5)	4a	0.6848(5)	-0.015(1)	0.219(1)	0.063
H(6)	4a	0.7875(7)	-0.128(1)	0.154(1)	0.062
H(7A)	4a	0.8926(6)	0.018(1)	0.177(1)	0.060
H(7B)	4a	0.8507(6)	0.005(1)	0.254(1)	0.060
H(8)	4a	0.7547(5)	0.141(1)	-0.0661(9)	0.054
H(9A)	4a	0.6729(6)	0.254(1)	-0.1782(9)	0.060
H(9B)	4a	0.7478(6)	0.298(1)	-0.1548(9)	0.060
H(10A)	4a	0.7490(7)	0.171(2)	-0.266(1)	0.076
H(10B)	4a	0.7971(7)	0.115(2)	-0.202(1)	0.076
H(11A)	4a	0.7408(7)	-0.048(1)	-0.252(1)	0.083
H(11B)	4a	0.6691(7)	0.022(1)	-0.247(1)	0.083

* Correspondence author (e-mail: rhett.kempe@ifok.uni-rostock.de)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(12A)	4a	0.7437(8)	-0.081(1)	-0.127(1)	0.085
H(12B)	4a	0.6697(8)	-0.123(1)	-0.154(1)	0.085
H(13A)	4a	0.6671(6)	-0.006(1)	-0.040(1)	0.058
H(13B)	4a	0.6196(6)	0.054(1)	-0.103(1)	0.058
H(14)	4a	0.7736(5)	0.385(1)	-0.0216(9)	0.047
H(15A)	4a	0.6958(8)	0.474(1)	-0.114(1)	0.074
H(15B)	4a	0.6569(8)	0.546(1)	-0.048(1)	0.074
H(16A)	4a	0.732(1)	0.687(2)	-0.105(1)	0.103
H(16B)	4a	0.794(1)	0.593(2)	-0.094(1)	0.103
H(17A)	4a	0.8062(7)	0.742(1)	0.004(1)	0.086
H(17B)	4a	0.7272(7)	0.732(1)	0.025(1)	0.086
H(18A)	4a	0.7870(7)	0.626(1)	0.117(1)	0.095
H(18B)	4a	0.8295(7)	0.548(1)	0.057(1)	0.095
H(19A)	4a	0.6862(6)	0.515(1)	0.0844(9)	0.065
H(19B)	4a	0.7442(6)	0.419(1)	0.1103(9)	0.065
H(20A)	4a	0.5947(6)	0.344(2)	-0.089(1)	0.072
H(20B)	4a	0.5611(6)	0.229(2)	-0.047(1)	0.072
H(21A)	4a	0.5765(8)	0.468(1)	0.019(1)	0.115
H(21B)	4a	0.5094(8)	0.429(1)	-0.024(1)	0.115
H(22A)	4a	0.469(1)	0.398(2)	0.080(1)	0.190
H(22B)	4a	0.541(1)	0.400(2)	0.119(1)	0.190
H(23A)	4a	0.4972(5)	0.172(1)	0.056(2)	0.106
H(23B)	4a	0.4626(5)	0.216(1)	0.132(2)	0.106
H(24)	4a	0.5709(6)	-0.065(1)	0.206(1)	0.058
H(25A)	4a	0.6273(6)	-0.088(1)	0.088(1)	0.073

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(25B)	4a	0.5556(6)	-0.072(1)	0.047(1)	0.073
H(26A)	4a	0.5758(7)	-0.296(1)	0.057(2)	0.117
H(26B)	4a	0.5897(7)	-0.284(1)	0.145(2)	0.117
H(27A)	4a	0.4614(6)	-0.250(1)	0.072(2)	0.118
H(27B)	4a	0.4785(6)	-0.358(1)	0.130(2)	0.118
H(28A)	4a	0.4743(7)	-0.222(1)	0.225(1)	0.108
H(28B)	4a	0.4056(7)	-0.212(1)	0.178(1)	0.108
H(29A)	4a	0.4482(6)	-0.008(1)	0.208(1)	0.074
H(29B)	4a	0.4452(6)	-0.026(1)	0.120(1)	0.074
H(30)	4a	0.6036(6)	0.156(1)	0.287(1)	0.089
H(31A)	4a	0.550(1)	0.389(2)	0.230(1)	0.090
H(31B)	4a	0.630(1)	0.353(2)	0.231(1)	0.090
H(32A)	4a	0.6274(9)	0.329(2)	0.370(1)	0.147
H(32B)	4a	0.6007(9)	0.466(2)	0.352(1)	0.147
H(33A)	4a	0.4955(8)	0.419(2)	0.377(1)	0.142
H(33B)	4a	0.5395(8)	0.360(2)	0.442(1)	0.142
H(34A)	4a	0.444(2)	0.250(2)	0.390(1)	0.239
H(34B)	4a	0.510(2)	0.177(2)	0.418(1)	0.239
H(35A)	4a	0.4567(7)	0.186(2)	0.270(1)	0.190
H(35B)	4a	0.4948(7)	0.071(2)	0.310(1)	0.190
F(1)	4a	0.3697(5)	0.0970(9)	-0.1600(9)	0.104(3)
F(2)	4a	0.4749(8)	0.174(2)	-0.146(1)	0.232(7)
F(3)	4a	0.3848(8)	0.258(2)	-0.078(1)	0.209(6)
F(4)	4a	0.425(1)	0.105(2)	-0.0604(7)	0.278(9)
B(1)	4a	0.4113(7)	0.170(2)	-0.1212(2)	0.20(1)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4a	0.7861(6)	0.039(2)	0.085(1)	0.040(7)	0.08(1)	0.05(1)	0.025(8)	0.008(6)	0.021(8)
C(2)	4a	0.8049(5)	0.164(1)	0.101(1)	0.027(4)	0.067(8)	0.04(1)	0.008(6)	-0.009(7)	0.029(9)
C(3)	4a	0.8180(6)	0.163(1)	0.190(1)	0.027(6)	0.073(9)	0.075(8)	-0.014(8)	-0.022(7)	-0.015(8)
C(4)	4a	0.7452(5)	0.145(1)	0.222(1)	0.046(6)	0.055(9)	0.049(7)	0.010(8)	-0.005(6)	-0.009(8)
C(5)	4a	0.7262(5)	0.025(1)	0.205(1)	0.022(6)	0.08(1)	0.058(8)	0.004(7)	-0.001(6)	0.038(7)
C(6)	4a	0.7865(7)	-0.035(1)	0.158(1)	0.055(8)	0.044(9)	0.056(8)	0.010(7)	0.006(7)	0.018(8)
C(7)	4a	0.8480(6)	0.030(1)	0.201(1)	0.045(7)	0.052(9)	0.053(8)	0.011(7)	-0.003(7)	0.008(6)
C(8)	4a	0.7077(5)	0.165(1)	-0.0824(9)	0.058(8)	0.038(7)	0.039(6)	-0.008(7)	-0.009(5)	-0.006(6)
C(9)	4a	0.7176(6)	0.225(1)	-0.1594(9)	0.073(8)	0.048(8)	0.028(6)	-0.010(6)	0.012(6)	-0.010(5)
C(10)	4a	0.7490(7)	0.131(2)	-0.216(1)	0.061(9)	0.09(2)	0.04(1)	0.01(1)	0.006(8)	-0.03(1)
C(11)	4a	0.7132(7)	0.010(1)	-0.221(1)	0.09(1)	0.07(1)	0.052(8)	0.006(9)	-0.007(8)	-0.022(8)
C(12)	4a	0.7000(8)	-0.050(1)	-0.147(1)	0.11(1)	0.032(7)	0.069(9)	-0.004(9)	0.01(1)	-0.016(7)
C(13)	4a	0.6674(6)	0.037(1)	-0.089(1)	0.064(8)	0.042(8)	0.039(6)	-0.014(6)	0.005(6)	-0.013(6)
C(14)	4a	0.7263(5)	0.410(1)	-0.0071(9)	0.047(6)	0.035(7)	0.035(6)	0.011(5)	-0.001(6)	0.008(5)
C(15)	4a	0.7018(8)	0.513(1)	-0.064(1)	0.11(1)	0.029(8)	0.051(7)	-0.014(8)	-0.009(9)	0.012(6)
C(16)	4a	0.751(1)	0.622(2)	-0.071(1)	0.19(2)	0.04(1)	0.023(8)	0.00(1)	0.03(1)	-0.007(7)
C(17)	4a	0.7673(7)	0.683(1)	0.009(1)	0.12(1)	0.035(9)	0.058(8)	-0.010(8)	0.030(9)	0.008(7)
C(18)	4a	0.7843(7)	0.585(1)	0.068(1)	0.12(1)	0.09(1)	0.032(7)	-0.06(1)	-0.016(8)	-0.002(7)
C(19)	4a	0.7312(6)	0.480(1)	0.0713(9)	0.086(8)	0.053(9)	0.024(5)	-0.009(7)	0.008(6)	-0.012(6)
C(20)	4a	0.5881(6)	0.306(2)	-0.040(1)	0.022(7)	0.10(2)	0.06(1)	0.027(8)	-0.017(7)	-0.018(9)
C(21)	4a	0.5464(8)	0.397(1)	0.008(1)	0.08(1)	0.05(1)	0.15(2)	0.034(8)	0.02(1)	0.01(1)
C(22)	4a	0.514(1)	0.360(2)	0.079(1)	0.28(2)	0.07(1)	0.13(2)	0.12(2)	0.10(2)	0.06(1)
C(23)	4a	0.5049(5)	0.221(1)	0.102(2)	0.038(6)	0.10(1)	0.13(1)	0.013(6)	0.00(1)	0.02(1)
C(24)	4a	0.5493(6)	-0.029(1)	0.161(1)	0.039(7)	0.026(7)	0.079(9)	-0.016(6)	0.013(7)	0.009(7)
C(25)	4a	0.5777(6)	-0.102(1)	0.093(1)	0.061(7)	0.033(7)	0.09(1)	-0.009(6)	0.050(9)	-0.033(7)
C(26)	4a	0.5627(7)	-0.251(1)	0.103(2)	0.093(9)	0.053(8)	0.15(1)	-0.007(7)	0.05(2)	0.02(1)
C(27)	4a	0.4862(6)	-0.269(1)	0.118(2)	0.083(9)	0.07(1)	0.14(1)	-0.039(8)	0.03(2)	0.02(2)
C(28)	4a	0.4553(7)	-0.195(1)	0.177(1)	0.064(9)	0.07(1)	0.13(1)	-0.039(8)	0.04(1)	-0.02(1)
C(29)	4a	0.4677(6)	-0.055(1)	0.166(1)	0.040(7)	0.07(1)	0.079(9)	-0.007(7)	0.021(7)	-0.020(8)
C(30)	4a	0.5661(6)	0.198(1)	0.259(1)	0.028(6)	0.023(8)	0.17(2)	-0.004(5)	0.032(8)	-0.032(8)
C(31)	4a	0.586(1)	0.340(2)	0.256(1)	0.10(1)	0.06(1)	0.06(1)	0.02(1)	0.00(1)	-0.01(1)
C(32)	4a	0.5896(9)	0.377(2)	0.347(1)	0.13(1)	0.13(2)	0.12(1)	-0.06(1)	0.05(1)	-0.08(1)
C(33)	4a	0.5282(8)	0.352(2)	0.388(1)	0.09(1)	0.14(2)	0.12(1)	0.01(1)	0.03(1)	-0.07(2)
C(34)	4a	0.493(2)	0.235(2)	0.379(1)	0.44(4)	0.11(2)	0.053(9)	-0.18(2)	0.05(2)	-0.02(1)
C(35)	4a	0.4964(7)	0.162(2)	0.301(1)	0.043(8)	0.33(3)	0.11(1)	0.06(2)	-0.011(9)	-0.10(2)
P(1)	4a	0.5743(1)	0.1426(3)	0.1555(8)	0.027(1)	0.036(2)	0.057(2)	-0.001(2)	0.012(2)	0.000(2)
P(2)	4a	0.6726(1)	0.2614(3)	-0.0031(8)	0.032(2)	0.036(2)	0.031(2)	0.007(1)	-0.005(1)	0.002(1)
Rh(1)	4a	0.68994(3)	0.15358(7)	0.1126(8)	0.0235(3)	0.0336(4)	0.0288(3)	0.0011(4)	0.0036(8)	0.0036(8)

2. 1,4-bis(dicyclohexylphosphino)-butane rhodium(I)-(Z,Z)-cycloocta-1,5-diene tetrafluoroborate, $C_{36}H_{64}BF_4P_2Rh$

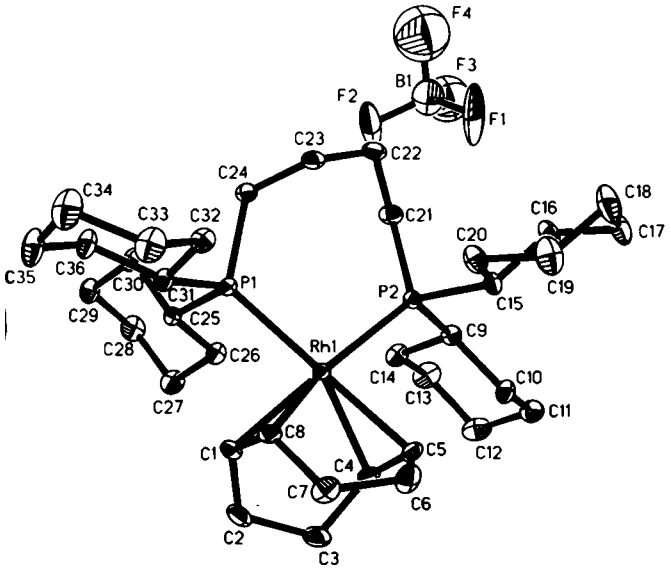


Table 4. Data collection and handling.

Crystal:	red prism, size 0.2 × 0.3 × 0.3 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.92 cm^{-1}
Diffractometer, scan mode:	STOE IPDS, 100 exposures, $\Delta\varphi$ = 2°
$2\theta_{max}$:	48.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10654, 5872
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4615
$N(param)_{refined}$:	387
Programs:	SHELXS-86 [4], SHELXL-93 [5]

Table 5. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.7327(3)	0.0742(5)	0.6108(2)	0.031
H(2A)	4e	0.6142(3)	0.1551(6)	0.5618(3)	0.048
H(2B)	4e	0.6529(3)	0.2900(6)	0.5540(3)	0.048
H(3A)	4e	0.5701(3)	0.2934(6)	0.6171(3)	0.054
H(3B)	4e	0.6429(3)	0.3867(6)	0.6451(3)	0.054
H(4)	4e	0.6262(3)	0.1589(5)	0.7189(2)	0.036
H(5)	4e	0.7279(3)	0.2322(4)	0.8168(3)	0.034
H(6A)	4e	0.8223(3)	0.3595(5)	0.8162(3)	0.049
H(6B)	4e	0.7558(3)	0.4606(5)	0.7716(3)	0.049
H(7A)	4e	0.7582(3)	0.4134(5)	0.6683(3)	0.042
H(7B)	4e	0.8451(3)	0.3928(5)	0.7219(3)	0.042
H(8)	4e	0.8335(3)	0.1612(4)	0.7031(2)	0.031
H(9)	4e	0.6444(2)	−0.0816(5)	0.8760(2)	0.029
H(10A)	4e	0.6556(3)	0.1451(5)	0.9036(3)	0.035
H(10B)	4e	0.6134(3)	0.1827(5)	0.8201(3)	0.035
H(11A)	4e	0.5462(3)	0.0463(5)	0.8994(3)	0.043
H(11B)	4e	0.5257(3)	0.1940(5)	0.8681(3)	0.043
H(12A)	4e	0.4716(3)	0.1131(5)	0.7504(3)	0.051
H(12B)	4e	0.4330(3)	0.0405(5)	0.7948(3)	0.051
H(13A)	4e	0.5073(3)	−0.1509(5)	0.8089(3)	0.051
H(13B)	4e	0.4640(3)	−0.1165(5)	0.7251(3)	0.051
H(14A)	4e	0.5726(3)	−0.0191(5)	0.7267(3)	0.038
H(14B)	4e	0.5935(3)	−0.1660(5)	0.7593(3)	0.038
H(15)	4e	0.7743(2)	0.1531(5)	0.9163(2)	0.030
H(16A)	4e	0.7499(3)	−0.0139(5)	0.9839(2)	0.040
H(16B)	4e	0.8259(3)	−0.0889(5)	0.9952(2)	0.040
H(17A)	4e	0.8498(3)	0.0400(6)	1.0972(3)	0.054
H(17B)	4e	0.8183(3)	0.1704(6)	1.0488(3)	0.054
H(18A)	4e	0.9534(3)	0.0320(7)	1.0709(3)	0.068
H(18B)	4e	0.9497(3)	0.1774(7)	1.1008(3)	0.068
H(19A)	4e	0.8978(3)	0.2664(6)	0.9839(3)	0.059
H(19B)	4e	0.9733(3)	0.1886(6)	0.9965(3)	0.059
H(20A)	4e	0.8733(3)	0.1343(5)	0.8822(3)	0.040

Table 5. Continued.

Atom	Site	x	y	z	U_{iso}
H(20B)	4e	0.9033(3)	0.0042(5)	0.9312(3)	0.040
H(21A)	4e	0.7356(3)	−0.2241(5)	0.8913(3)	0.035
H(21B)	4e	0.7205(3)	−0.2507(5)	0.8105(3)	0.035
H(22A)	4e	0.8304(3)	−0.3540(5)	0.8852(2)	0.038
H(22B)	4e	0.8638(3)	−0.2200(5)	0.9288(2)	0.038
H(23A)	4e	0.9185(2)	−0.2779(5)	0.8498(2)	0.034
H(23B)	4e	0.8842(2)	−0.1308(5)	0.8365(2)	0.034
H(24A)	4e	0.7795(3)	−0.3166(4)	0.7503(2)	0.032
H(24B)	4e	0.8525(3)	−0.3139(4)	0.7355(2)	0.032
H(25)	4e	0.7018(2)	−0.1333(4)	0.5711(2)	0.025
H(26A)	4e	0.6268(2)	−0.2025(5)	0.6579(2)	0.033
H(26B)	4e	0.6255(2)	−0.0568(5)	0.6260(2)	0.033
H(27A)	4e	0.5140(3)	−0.1763(5)	0.5516(3)	0.043
H(27B)	4e	0.5606(3)	−0.1383(5)	0.5074(3)	0.043
H(28A)	4e	0.5234(3)	−0.3617(5)	0.4843(3)	0.045
H(28B)	4e	0.5609(3)	−0.3943(5)	0.5685(3)	0.045
H(29A)	4e	0.6423(3)	−0.3215(5)	0.4882(3)	0.039
H(29B)	4e	0.6421(3)	−0.4667(5)	0.5199(3)	0.039
H(30A)	4e	0.7541(3)	−0.3511(5)	0.5957(3)	0.035
H(30B)	4e	0.7056(3)	−0.3838(5)	0.6388(3)	0.035
H(31)	4e	0.8437(2)	0.0008(5)	0.6354(2)	0.030
H(32A)	4e	0.9252(2)	0.0231(5)	0.7595(3)	0.034
H(32B)	4e	0.9542(2)	−0.1262(5)	0.7609(3)	0.034
H(33A)	4e	1.0401(3)	0.0212(6)	0.7478(3)	0.053
H(33B)	4e	0.9739(3)	0.0877(6)	0.6789(3)	0.053
H(34A)	4e	1.0392(3)	−0.0601(7)	0.6386(3)	0.066
H(34B)	4e	1.0293(3)	−0.1786(7)	0.6854(3)	0.066
H(35A)	4e	0.9415(3)	−0.2094(7)	0.5666(3)	0.060
H(35B)	4e	0.9110(3)	−0.0606(7)	0.5620(3)	0.060
H(36A)	4e	0.8909(3)	−0.2698(5)	0.6465(3)	0.041
H(36B)	4e	0.8252(3)	−0.2058(5)	0.5763(3)	0.041
F(3)	4e	0.6460(4)	−0.5736(7)	0.9438(4)	0.166(3)
F(4)	4e	0.7520(5)	−0.612(1)	0.9644(6)	0.234(4)

Table 6. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.7273(3)	0.1607(5)	0.6263(2)	0.031(3)	0.026(3)	0.022(2)	0.003(2)	0.013(2)	0.008(2)
C(2)	4*e*	0.6515(3)	0.2242(6)	0.5890(3)	0.041(3)	0.051(3)	0.024(3)	0.014(3)	0.011(2)	0.015(2)
C(3)	4*e*	0.6259(3)	0.2930(6)	0.6394(3)	0.042(3)	0.054(4)	0.040(3)	0.028(3)	0.018(3)	0.020(3)
C(4)	4*e*	0.6542(3)	0.2304(5)	0.7128(2)	0.036(3)	0.024(2)	0.035(3)	0.014(2)	0.021(2)	0.008(2)
C(5)	4*e*	0.7187(3)	0.2709(4)	0.7719(3)	0.044(3)	0.015(2)	0.035(3)	0.005(2)	0.026(2)	0.002(2)
C(6)	4*e*	0.7753(3)	0.3700(5)	0.7714(3)	0.061(4)	0.024(3)	0.049(3)	–0.011(3)	0.034(3)	–0.007(2)
C(7)	4*e*	0.7938(3)	0.3576(5)	0.7080(3)	0.037(3)	0.025(3)	0.050(3)	–0.005(2)	0.027(3)	0.001(2)
C(8)	4*e*	0.7899(3)	0.2153(4)	0.6809(2)	0.032(3)	0.018(2)	0.034(3)	0.004(2)	0.022(2)	0.010(2)
C(9)	4*e*	0.6401(2)	–0.0189(5)	0.8369(2)	0.024(2)	0.026(2)	0.029(3)	–0.002(2)	0.016(2)	0.002(2)
C(10)	4*e*	0.6168(3)	0.1154(5)	0.8563(3)	0.025(2)	0.035(3)	0.029(3)	0.000(2)	0.014(2)	–0.008(2)
C(11)	4*e*	0.5410(3)	0.1049(5)	0.8591(3)	0.034(3)	0.041(3)	0.043(3)	0.007(2)	0.026(3)	0.007(2)
C(12)	4*e*	0.4806(3)	0.0500(5)	0.7900(3)	0.024(3)	0.045(3)	0.063(4)	0.005(2)	0.023(3)	0.007(3)
C(13)	4*e*	0.5033(3)	–0.0853(5)	0.7718(3)	0.025(3)	0.043(3)	0.061(4)	–0.009(2)	0.020(3)	–0.004(3)
C(14)	4*e*	0.5784(3)	–0.0762(5)	0.7677(3)	0.024(2)	0.031(3)	0.039(3)	–0.003(2)	0.013(2)	–0.006(2)
C(15)	4*e*	0.7972(2)	0.0634(5)	0.9190(2)	0.023(2)	0.034(3)	0.018(2)	0.000(2)	0.010(2)	–0.001(2)
C(16)	4*e*	0.8015(3)	–0.0006(5)	0.9884(2)	0.034(3)	0.048(3)	0.021(3)	0.000(2)	0.015(2)	0.006(2)
C(17)	4*e*	0.8456(3)	0.0859(6)	1.0533(3)	0.045(3)	0.067(4)	0.022(3)	–0.001(3)	0.013(2)	–0.004(3)
C(18)	4*e*	0.9239(3)	0.1155(7)	1.0602(3)	0.045(3)	0.091(5)	0.026(3)	–0.011(3)	0.008(3)	–0.016(3)
C(19)	4*e*	0.9214(3)	0.1774(6)	0.9913(3)	0.036(3)	0.070(4)	0.040(3)	–0.021(3)	0.015(3)	–0.014(3)
C(20)	4*e*	0.8767(3)	0.0898(5)	0.9261(3)	0.027(3)	0.048(3)	0.026(3)	–0.006(2)	0.014(2)	0.001(2)
C(21)	4*e*	0.7530(3)	–0.2013(5)	0.8545(3)	0.030(3)	0.026(3)	0.035(3)	0.001(2)	0.018(2)	0.009(2)
C(22)	4*e*	0.8324(3)	–0.2563(5)	0.8804(2)	0.033(3)	0.029(3)	0.029(3)	0.008(2)	0.011(2)	0.010(2)
C(23)	4*e*	0.8711(2)	–0.2265(5)	0.8325(2)	0.023(2)	0.031(3)	0.029(3)	0.008(2)	0.008(2)	0.005(2)
C(24)	4*e*	0.8221(3)	–0.2605(4)	0.7534(2)	0.027(2)	0.023(2)	0.027(2)	0.004(2)	0.010(2)	0.002(2)
C(25)	4*e*	0.7031(2)	–0.1824(4)	0.6136(2)	0.022(2)	0.022(2)	0.020(2)	0.002(2)	0.010(2)	0.000(2)
C(26)	4*e*	0.6285(2)	–0.1529(5)	0.6173(2)	0.022(2)	0.030(3)	0.028(3)	–0.002(2)	0.009(2)	–0.006(2)
C(27)	4*e*	0.5613(3)	–0.1931(5)	0.5474(3)	0.020(2)	0.043(3)	0.039(3)	0.000(2)	0.008(2)	–0.006(2)
C(28)	4*e*	0.5657(3)	–0.3392(5)	0.5311(3)	0.025(3)	0.044(3)	0.033(3)	–0.011(2)	0.003(2)	–0.011(2)
C(29)	4*e*	0.6396(3)	–0.3704(5)	0.5285(3)	0.035(3)	0.033(3)	0.030(3)	–0.007(2)	0.012(2)	–0.012(2)
C(30)	4*e*	0.7065(3)	–0.3312(5)	0.5988(3)	0.027(3)	0.028(3)	0.034(3)	–0.003(2)	0.015(2)	–0.010(2)
C(31)	4*e*	0.8595(2)	–0.0815(5)	0.6654(2)	0.023(2)	0.029(2)	0.026(2)	0.000(2)	0.014(2)	–0.002(2)
C(32)	4*e*	0.9342(2)	–0.0465(5)	0.7303(3)	0.021(2)	0.031(3)	0.032(3)	–0.002(2)	0.009(2)	–0.003(2)
C(33)	4*e*	0.9918(3)	0.0032(6)	0.7054(3)	0.025(3)	0.056(4)	0.054(4)	–0.010(3)	0.020(3)	–0.012(3)
C(34)	4*e*	1.0049(3)	–0.0985(7)	0.6570(3)	0.030(3)	0.078(5)	0.064(4)	–0.011(3)	0.027(3)	–0.017(3)
C(35)	4*e*	0.9315(3)	–0.1380(7)	0.5942(3)	0.041(3)	0.072(4)	0.052(4)	–0.006(3)	0.034(3)	–0.018(3)
C(36)	4*e*	0.8731(3)	–0.1865(5)	0.6188(3)	0.026(3)	0.046(3)	0.035(3)	–0.002(2)	0.017(2)	–0.014(2)
B(1)	4*e*	0.7013(4)	–0.5178(5)	0.9333(2)	0.117(8)	0.056(5)	0.087(7)	–0.017(6)	0.059(6)	0.001(5)
F(1)	4*e*	0.7288(4)	–0.4072(5)	0.9703(3)	0.266(7)	0.103(4)	0.108(4)	–0.106(4)	0.137(5)	–0.079(3)
F(2)	4*e*	0.6883(3)	–0.5103(5)	0.8649(2)	0.165(5)	0.109(4)	0.052(2)	–0.026(3)	0.063(3)	–0.025(2)
P(1)	4*e*	0.78405(6)	–0.1150(1)	0.69438(6)	0.0178(6)	0.0184(6)	0.0208(6)	0.0009(4)	0.0088(5)	–0.0011(4)
P(2)	4*e*	0.73429(6)	–0.0228(1)	0.83452(6)	0.0188(6)	0.0215(6)	0.0192(6)	–0.0004(5)	0.0099(5)	0.0024(5)
Rh(1)	4*e*	0.74158(2)	0.07564(3)	0.73207(2)	0.0184(2)	0.0157(2)	0.0178(2)	0.0010(1)	0.0093(1)	0.0016(1)

Acknowledgment. We thank the Fonds der Chemischen Industrie.

References

1. Schrock, R. R.; Osborn, J. A.: Preparation and Properties of Some Cationic Complexes of Rhodium(I) and Rhodium(III). J. Am. Chem. Soc. **93** (1971) 2397-2407.
2. Heller, D.; Borns, S.; Baumann, W.; Selke, R.: Kinetic investigations of the hydrogenation of diolefin ligands in catalyst precursors for the asymmetric reduction of prochiral olefins, (II). Chem. Ber. **129** (1996) 85-89.
3. Anderson, M. P.; Pignolet, L. H.: Rhodium Complexes of 1,4-Bis(diphenylphosphino)butane. Crystal and Molecular Structures of $[Rh(dppb)_2]BF_4 \cdot C_4H_{10}O$ and $[Rh(cod)(dppb)]BF_4$. Inorg. Chem. **20** (1981) 4101-4107.
4. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct Methods for Large Structures. Acta Crystallogr. A**46** (1999) 467-473.
5. Sheldrick, G. M.: SHELXL-93. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1993.