Crystal structure of (η^4-cycloocta-1,5-dien)(1,2-bis(diethylphosphino)-ethane)rhodium(I) tetrafluoroborate, $[Rh(C_8H_{12})(C_{10}H_{24}P_2)](BF_4)$

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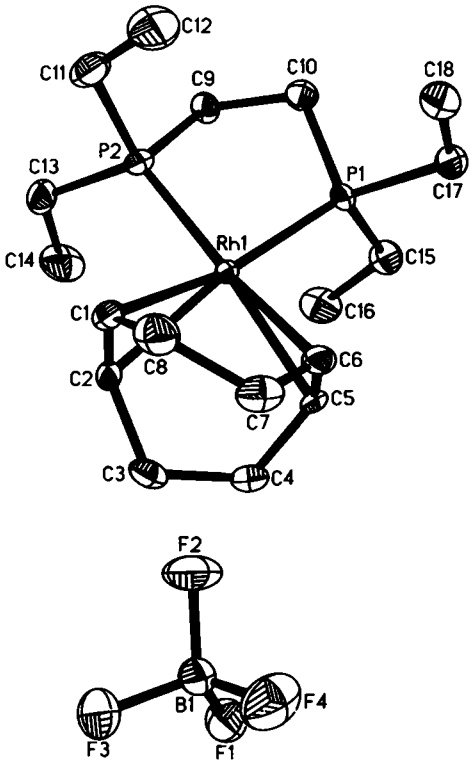

Abstract

$C_{18}H_{36}BF_4P_2Rh$, monoclinic, $P12_1/n1$ (no. 14), $a = 15.522(3)$ Å, $b = 9.173(2)$ Å, $c = 15.862(3)$ Å, $\beta = 103.91(3)°$, $V = 2192.3$ Å^3, $Z = 4$, $R_{gt}(F) = 0.037$, $wR_{obs}(F^2) = 0.087$, $T = 200$ K.

Source of material

Standard preparation according to [1]. The ligand is commercially available.

Discussion

For chelating phosphane ligands it is known that a growing sterical hindrance of the functional groups at the phosphorus atom accompanies a distortion towards tetrahedral geometry (for phenyl groups see [2,3], for cyclohexyl groups [4]). On the other hand the ideal square planar arrangement results, as expected, for sterically less demanding groups. Accordingly in the title compound, the small ethyl group causes a dihedral angle between the planes P,Rh,P and X,Rh,X (X = centroid of the double bond) of only 0.7°.

Table 1. Data collection and handling.

Crystal:	orange needle, size 0.1 × 0.1 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.58 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS I, φ
$2\theta_{max}$:	48.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6293, 3290
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2491
$N(param)_{refined}$:	235
Programs:	SHELXS-86 [5], SHELXL-97 [6]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.3267	–0.0358	0.5339	0.029
H(2)	4e	0.1783	–0.0616	0.4833	0.029
H(3A)	4e	0.1154	–0.1620	0.3481	0.030
H(3B)	4e	0.2074	–0.1482	0.3202	0.030
H(4A)	4e	0.1229	0.0170	0.2331	0.029
H(4B)	4e	0.0730	0.0662	0.3055	0.029
H(5)	4e	0.1643	0.2629	0.3165	0.022
H(6)	4e	0.3129	0.2594	0.3535	0.027
H(7A)	4e	0.3923	0.0680	0.3108	0.034
H(7B)	4e	0.3055	–0.0325	0.2897	0.034
H(8A)	4e	0.3905	–0.1382	0.4099	0.040
H(8B)	4e	0.4246	0.0145	0.4538	0.040
H(9A)	4e	0.1334	0.3557	0.6191	0.030
H(9B)	4e	0.2210	0.3782	0.6957	0.030
H(10A)	4e	0.2930	0.5030	0.6064	0.032
H(10B)	4e	0.1943	0.5683	0.5812	0.032
H(11A)	4e	0.3477	0.1959	0.7387	0.039
H(11B)	4e	0.3636	0.0524	0.6877	0.039
H(12A)	4e	0.4835	0.2109	0.7005	0.073
H(12B)	4e	0.4190	0.3365	0.6515	0.073
H(12C)	4e	0.4348	0.1927	0.6000	0.073
H(13A)	4e	0.2022	–0.0504	0.6442	0.035
H(13B)	4e	0.1813	0.0681	0.7104	0.035
H(14A)	4e	0.0483	–0.0290	0.6268	0.065
H(14B)	4e	0.0739	0.0221	0.5394	0.065
H(14C)	4e	0.0530	0.1410	0.6056	0.065
H(15A)	4e	0.0956	0.5782	0.4336	0.031
H(15B)	4e	0.1058	0.4942	0.3484	0.031
H(16A)	4e	–0.0250	0.4235	0.3843	0.053
H(16B)	4e	0.0297	0.3704	0.4780	0.053
H(16C)	4e	0.0400	0.2861	0.3926	0.053
H(17A)	4e	0.2756	0.5340	0.3618	0.031
H(17B)	4e	0.2755	0.6451	0.4393	0.031
H(18A)	4e	0.4219	0.5886	0.4354	0.049
H(18B)	4e	0.4035	0.4191	0.4470	0.049
H(18C)	4e	0.4035	0.5306	0.5246	0.049

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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)	4*e*	0.3051(3)	–0.0458(5)	0.4728(3)	0.037(3)	0.017(3)	0.016(3)	0.011(2)	–0.002(2)	–0.002(2)
C(2)	4*e*	0.2136(4)	–0.0666(5)	0.4420(3)	0.047(3)	0.013(3)	0.013(3)	0.000(2)	0.009(3)	0.000(2)
C(3)	4*e*	0.1662(3)	–0.0963(5)	0.3487(3)	0.027(3)	0.021(3)	0.029(3)	–0.009(2)	0.008(2)	–0.006(2)
C(4)	4*e*	0.1317(3)	0.0402(5)	0.2955(3)	0.023(2)	0.029(3)	0.018(3)	–0.002(2)	0.000(2)	–0.005(2)
C(5)	4*e*	0.1921(3)	0.1706(5)	0.3166(3)	0.027(2)	0.018(2)	0.007(3)	–0.001(2)	0.001(2)	0.000(2)
C(6)	4*e*	0.2833(3)	0.1698(6)	0.3361(3)	0.027(2)	0.026(3)	0.016(3)	–0.010(2)	0.007(2)	–0.004(2)
C(7)	4*e*	0.3400(3)	0.0378(6)	0.3320(3)	0.023(3)	0.038(3)	0.026(3)	–0.003(2)	0.011(2)	–0.010(2)
C(8)	4*e*	0.3718(3)	–0.0379(6)	0.4200(4)	0.034(3)	0.027(3)	0.035(4)	0.008(2)	0.003(3)	–0.006(2)
C(9)	4*e*	0.1990(3)	0.3583(5)	0.6328(3)	0.041(3)	0.019(3)	0.015(3)	0.002(2)	0.009(3)	–0.006(2)
C(10)	4*e*	0.2300(3)	0.4793(5)	0.5801(3)	0.032(3)	0.018(3)	0.028(3)	0.001(2)	0.005(3)	–0.006(2)
C(11)	4*e*	0.3511(3)	0.1582(6)	0.6811(4)	0.035(3)	0.036(3)	0.023(3)	0.003(3)	–0.001(3)	0.001(3)
C(12)	4*e*	0.4290(4)	0.2310(7)	0.6561(5)	0.027(3)	0.052(4)	0.056(5)	0.000(3)	–0.010(3)	0.003(3)
C(13)	4*e*	0.1767(4)	0.0474(6)	0.6482(3)	0.047(3)	0.024(3)	0.019(3)	0.001(2)	0.012(3)	0.002(2)
C(14)	4*e*	0.0793(4)	0.0452(7)	0.6008(4)	0.037(3)	0.048(4)	0.047(4)	–0.019(3)	0.017(3)	–0.007(3)
C(15)	4*e*	0.1066(3)	0.4814(5)	0.4106(3)	0.027(3)	0.022(3)	0.029(3)	0.007(2)	0.006(2)	0.001(2)
C(16)	4*e*	0.0312(3)	0.3816(6)	0.4169(4)	0.020(3)	0.037(3)	0.045(4)	0.003(2)	0.000(3)	–0.004(3)
C(17)	4*e*	0.2895(3)	0.5437(5)	0.4258(3)	0.033(3)	0.017(3)	0.025(3)	0.001(2)	0.004(3)	0.002(2)
C(18)	4*e*	0.3884(3)	0.5182(6)	0.4614(4)	0.031(3)	0.032(3)	0.032(3)	–0.006(2)	0.004(3)	0.001(3)
B(1)	4*e*	0.4330(4)	–0.2483(6)	0.6878(4)	0.029(3)	0.023(3)	0.028(4)	0.001(3)	0.002(3)	0.000(3)
F(1)	4*e*	0.3415(2)	–0.2246(3)	0.6700(2)	0.030(2)	0.035(2)	0.046(2)	0.004(1)	0.001(2)	0.003(2)
F(2)	4*e*	0.4743(2)	–0.1764(4)	0.7636(2)	0.052(2)	0.052(2)	0.040(2)	–0.010(2)	–0.006(2)	–0.016(2)
F(3)	4*e*	0.4474(2)	–0.3971(4)	0.6977(2)	0.061(2)	0.031(2)	0.053(3)	0.016(2)	–0.004(2)	0.001(2)
F(4)	4*e*	0.4643(2)	–0.1979(4)	0.6188(2)	0.050(2)	0.081(3)	0.043(2)	–0.017(2)	0.013(2)	0.012(2)
P(1)	4*e*	0.21792(8)	0.4203(1)	0.46805(8)	0.0207(6)	0.0111(6)	0.0187(7)	–0.0011(5)	0.0025(6)	0.0015(5)
P(2)	4*e*	0.24197(8)	0.1819(1)	0.60603(8)	0.0204(6)	0.0202(7)	0.0143(7)	–0.0002(6)	0.0001(5)	–0.0002(6)
Rh(1)	4*e*	0.24006(2)	0.17505(4)	0.46177(2)	0.0188(2)	0.0109(2)	0.0107(2)	0.0006(2)	0.0015(1)	–0.0001(2)

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