Inorganica Chimica Acta, 57 (1982) 1–7

The Crystal and Molecular Structure of (Cycloocta-1,5-diene)(η^5-triphenylphosphonium Cyclopentadienylide)rhodium(I) Tetraphenylborate Salt

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Received July 20, 1981

The structure of the title compound has been determined by counter data. The crystals are triclinic, space group $P\bar{1}$ *with* $a = 14.912(5)$, $b = 12.973(4)$, $c = 11.696(4)$ Å, $\alpha = 105.9(1)°$, $\beta = 81.2(1)°$, $\gamma = 99.4(1)°$, $Z = 2$. *The structure has been refined by full-matrix least-squares to a final conventional R value of 0.076. The rhodium atom is bonded to the five carbon atoms of the planar cyclopentadienyl ring (Rh–C 2.22–2.29 Å) and to the four olefinic carbon atoms (Rh–C 2.13–2.28 Å) of the cycloocta-1,5-diene. The geometry around the rhodium atom may be described as trigonal. Some close contacts are observed between the rhodium atom and the diene hydrogen atoms (Rh–H(49) 2.28 Å; Rh--H(48) 2.52 Å; Rh--H(52) 2.39 Å; Rh--H(53) 2.32 Å).*

Introduction

Transition metal complexes containing trialkyl- or triaryl phosphonium cyclopentadienylide or dialkyl-sulphonium cyclopentadienylide are interesting compounds in that they make possible studies of the effect of coordination at a transition metal on the properties of cyclopentadienyl ion.

Although in the fundamental state $Ph_3PC_5H_4$ and $Me_2SC_5H_4$ are considered to be the resonance hybrids of ylidic and ylenic structures [1], the contribution of the former being dominant, coordination of the species at the metal occurs in the ylidic form [2]. Therefore, the five-membered ring carbon of the cyclopentadienylide acts as a six π-electron donor, similar to the cyclopentadienyl ion. Information on the coordination effect at cyclopentadienylide has been obtained by ^1H and ^{13}C NMR spectra [2]. To our knowledge only the compounds $(Me_2SC_5H_4)Cr(CO)_3$ and $[(Ph_3PC_5H_4)Co(CO)_2][Co(CO)_4]$ have been studied by X-ray diffraction [3, 4].

Recently some of us have reported [5] the synthesis and ^1H and ^{13}C NMR of (η^5-triphenylphosphonium cyclopentadienylide)rhodium(I) and rhodium(III) complexes. The ^{13}C chemical shift values were in all cases very similar to those reported for analogous complexes [2]. For the substrates $[Rh(Ph_3PC_5H_4)(1,5\text{-}C_8H_{12})]PF_6$ and $[Rh(Ph_3PC_5H_4)(C_7H_8)]PF_6$ (C_8H_{12} = cycloocta-1,5-diene; C_7H_8 = norbornadiene) the ^1H NMR spectra show a difference in the chemical shift between the α and β protons of $Ph_3PC_5H_4$ significantly larger than that usually found for other complexes containing the triphenylphosphonium cyclopentadienylide [6].

In order to assist the interpretation of the NMR spectra we wished to undertake an X-ray structure determination of one representative complex. Unfortunately suitable crystals can be obtained only by using tetraphenylborate as the counterion; the resulting compounds however were not soluble enough to obtain reliable NMR data.

The X-ray structure determination of the title compound is here reported in order to also elucidate the influence of triphenylphosphonium cyclopentadienylide on the bonding to the rhodium atom of the coordinated diolefin [7].

Experimental

$[Rh(Ph_3PC_5H_4)(1,5\text{-}C_8H_{12})]BPh_4$ was prepared by adding $Na[BPh_4]$ dissolved in methanol to a solution of the corresponding hexafluorophosphate salt [5] in the same solvent. Yellow-brown crystals suitable for X-ray diffraction were obtained by slow evaporation of a dichloromethane solution.

Crystal data: $C_{55}H_{51}PBRh$, $M = 856.7$, triclinic, $a = 14.912(5)$, $b = 12.973(4)$, $c = 11.696(4)$ Å, $\alpha = 105.9(1)°$, $\beta = 81.2(1)°$, $\gamma = 99.4(1)°$, $U = 2127.6$, $D_c = 1.34$ g cm^{-3}, $Z = 2$, F(000) = 892. Space group $P\bar{1}$, Mo K_α radiation, $\lambda = 0.7107$ Å, $\mu(MoK_\alpha) = 4.12$

TABLE I. Fractional Coordinates ($\times 10^4$), with e.s.d.s in Parentheses and isotropic U ($\times 10^3$).

	x	y	z	U		x	y	z
Rh	844(1)	2831(1)	3581(1)	–				
C(43)	442(11)	2673(16)	5440(14)	–	H(43)	–154	2811	5811
C(44)	686(12)	1668(14)	4785(15)	–	H(44)	296	998	4664
C(45)	1607(10)	1823(12)	4348(12)	–	H(45)	1950	1277	3849
C(46)	1941(9)	2920(11)	4766(11)	–				
C(47)	1201(11)	3436(13)	5454(13)	–	H(47)	1225	4182	5862
C(48)	1482(15)	3271(15)	1852(19)	–	H(48)	2076	3471	2102
C(49)	1167(15)	2246(16)	1702(12)	–	H(49)	1532	1782	1875
C(50)	260(20)	1777(18)	1255(21)	–	H(501)	121	1075	1393
					H(502)	335	1716	419
C(51)	–538(15)	2357(22)	1770(22)	–	H(511)	–546	2898	1364
					H(512)	–1077	1853	1666
C(52)	–496(14)	2842(23)	3061(24)	–	H(52)	–780	2446	3623
C(53)	–85(13)	3878(16)	3465(15)	–	H(53)	–125	4158	4309
C(54)	397(16)	4563(16)	2776(20)	–	H(541)	–51	4850	2499
					H(542)	736	5131	3304
C(55)	1043(11)	4173(14)	1728(16)	–	H(551)	724	3926	1040
					H(552)	1505	4750	1628
P	3077(3)	3558(3)	4699(3)	–				
C(1)	3042(6)	4982(8)	5234(7)	39(4)				
C(2)	2671(6)	5514(8)	4564(7)	49(4)	H(2)	2643	5124	3819
C(3)	2606(6)	6621(8)	4989(7)	58(5)	H(3)	2354	6983	4532
C(4)	2912(6)	7194(8)	6083(7)	56(5)	H(4)	2868	7948	6372
C(5)	3283(6)	6662(8)	6753(7)	62(5)	H(5)	3491	7052	7498
C(6)	3348(6)	5556(8)	6328(7)	47(4)	H(6)	3601	5193	6785
C(7)	3685(6)	3280(7)	3228(10)	40(4)				
C(8)	3537(6)	2270(7)	2418(10)	67(5)	H(8)	3105	1713	2643
C(9)	4021(6)	2078(7)	1278(10)	71(5)	H(9)	3920	1390	726
C(10)	4654(6)	2895(7)	947(10)	69(6)	H(10)	4984	2764	171
C(11)	4803(6)	3905(7)	1757(10)	69(5)	H(11)	5235	4461	1533
C(12)	4319(6)	4097(7)	2898(10)	49(4)	H(12)	4420	4784	3449
C(13)	3672(5)	3114(8)	5663(8)	33(4)				
C(14)	3203(5)	2981(8)	6751(8)	50(4)	H(14)	2585	3119	6959
C(15)	3642(5)	2644(8)	7532(8)	58(5)	H(15)	3323	2553	8272
C(16)	4549(5)	2440(8)	7225(8)	56(5)	H(16)	4848	2211	7757
C(17)	5018(5)	2574(8)	6138(8)	63(5)	H(17)	5636	2435	5929
C(18)	4579(5)	2911(8)	5357(8)	56(5)	H(18)	4898	3001	4616
B	7764(11)	1669(13)	–1430(14)	–				
C(19)	7404(6)	2870(8)	–994(8)	43(4)				
C(20)	7599(6)	3555(8)	111(8)	52(4)	H(20)	7952	3337	595
C(21)	7276(6)	4561(8)	506(8)	67(6)	H(21)	7408	5028	1259
C(22)	6757(6)	4880(8)	–204(8)	65(5)	H(22)	6537	5565	65
C(23)	6563(6)	4195(8)	–1309(8)	70(6)	H(23)	6210	4412	–1793
C(24)	6886(6)	3189(8)	–1704(8)	54(4)	H(24)	6754	2722	–2457
C(25)	7364(5)	971(7)	–2721(9)	43(4)				
C(26)	7876(5)	1018(7)	–3812(9)	51(4)	H(26)	8482	1386	–3827
C(27)	7499(5)	527(7)	–4880(9)	55(5)	H(27)	7848	560	–5623
C(28)	6609(5)	–12(7)	–4858(9)	75(6)	H(28)	6352	–347	–5585
C(29)	6096(5)	–60(7)	–3767(9)	74(5)	H(29)	5490	–427	–3752
C(30)	6474(5)	432(7)	–2698(9)	63(5)	H(30)	6125	399	–1956
C(31)	7367(5)	1019(8)	–334(8)	44(4)				
C(32)	7889(5)	376(8)	10(8)	53(4)	H(32)	8489	283	–377
C(33)	7529(5)	–130(8)	921(8)	57(5)	H(33)	7884	–568	1156
C(34)	6647(5)	7(8)	1489(8)	70(5)	H(34)	6401	–338	2109
C(35)	6126(5)	649(8)	1145(8)	59(5)	H(35)	5525	742	1531
C(36)	6486(5)	1155(8)	233(8)	54(5)	H(36)	6131	1593	–1
C(37)	8921(6)	1785(6)	–1666(8)	36(4)				

(continued on facing page)

TABLE I. *(continued)*

	x	y	z	U		x	y	z
C(38)	9457(6)	2789(6)	−1374(8)	48(4)	H(38)	9174	3429	−1043
C(39)	1.0409(6)	2854(6)	−1570(8)	58(5)	H(39)	1.0773	3539	−1372
C(40)	1.0824(6)	1915(6)	−2057(8)	57(5)	H(40)	1.1472	1959	−2190
C(41)	1.0289(6)	910(6)	−2348(8)	60(5)	H(41)	1.0572	270	−2679
C(42)	9337(6)	845(6)	−2152(8)	49(4)	H(42)	8972	161	−2351

TABLE II. Anisotropic Thermal Parameters ($\times 10^3$) in the Form $\exp[-2\pi^2 \Sigma U_{ij} a_i^* a_j^* h_i h_j]$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Rh	29.8(7)	44.6(8)	30.8(7)	13.4(6)	−3.1(5)	2.7(5)
C(43)	33(11)	82(15)	47(12)	20(11)	1(9)	4(11)
C(44)	65(14)	42(12)	64(13)	36(11)	−6(11)	−20(10)
C(45)	58(11)	37(11)	35(9)	18(4)	−13(8)	10(9)
C(46)	38(9)	22(9)	32(8)	6(7)	−4(7)	12(7)
C(47)	33(11)	49(12)	49(11)	12(9)	0(9)	2(10)
C(48)	80(19)	57(12)	151(23)	−23(13)	−61(16)	37(13)
C(49)	155(20)	50(15)	26(9)	−7(9)	−38(11)	45(14)
C(50)	205(30)	81(19)	108(21)	38(17)	20(23)	−31(20)
C(51)	102(19)	239(30)	100(23)	106(22)	−93(17)	−73(18)
C(52)	65(16)	190(27)	156(25)	111(22)	−4(15)	0(16)
C(53)	78(14)	86(15)	53(11)	46(12)	3(10)	17(12)
C(54)	170(24)	75(18)	129(22)	−4(16)	28(19)	20(16)
C(55)	54(12)	89(14)	68(15)	35(12)	7(11)	25(11)
P	40(3)	35(3)	39(3)	10(2)	−9(2)	3(2)
B	32(11)	41(11)	24(10)	8(8)	5(8)	−1(9)

TABLE III. Selected Interatomic Distances (Å) and Angles (°), with e.s.d.s. in Parentheses.

Bond Distances			
Rh–C(43)	2.22(1)	Rh–C(48)	2.28(2)
Rh–C(44)	2.29(1)	Rh–C(49)	2.13(1)
Rh–C(45)	2.29(1)	Rh–C(52)	2.18(2)
Rh–C(46)	2.27(1)	Rh–C(53)	2.13(1)
Rh–C(47)	2.23(1)	Rh–C(48,47)	2.10(2)
		Rh–C(52,53)	2.04(2)
Av of 5 Rh–C	2.26		
		C(48)–C(49)	1.31(2)
C(43)–C(44)	1.39(1)	C(49)–C(50)	1.50(1)
C(44)–C(45)	1.39(1)	C(50)–C(51)	1.47(1)
C(45)–C(46)	1.40(1)	C(51)–C(52)	1.48(2)
C(46)–C(47)	1.41(1)	C(52)–C(53)	1.37(2)
C(43)–C(47)	1.37(1)	C(53)–C(54)	1.40(2)
		C(54)–C(55)	1.46(2)
Av of 5 C–C	1.39	C(48)–C(55)	1.48(2)
P–C(46)	1.76(1)	P–C(1)	1.78(1)
P–C(7)	1.79(1)	P–C(13)	1.79(1)
B–C(19)	1.65(1)	B–C(25)	1.68(1)
B–C(31)	1.70(1)	B–C(37)	1.69(1)

(continued overleaf)

TABLE III. *(continued)*

Angles			
C(43)–C(44)–C(45)	107(1)	C(48)–C(49)–C(50)	122(1)
C(44)–C(45)–C(46)	108(1)	C(49)–C(50)–C(51)	119(2)
C(45)–C(46)–C(47)	107(1)	C(50)–C(51)–C(52)	110(1)
C(46)–C(47)–C(43)	108(1)	C(51)–C(52)–C(53)	121(2)
C(47)–C(43)-C(44)	109(1)	C(52)–C(53)–C(54)	127(1)
		C(53)–C(54)–C(55)	122(1)
		C(54)–C(55)–C(48)	112(1)
		C(55)–C(48)–C(49)	130(1)
C(45)–C(46)–P	129.2(8)	C(19)–B–C(25)	109.9(7)
C(47)–C(46)–P	123.4(8)	C(19)–B–C(31)	107.4(7)
C(46)–P–C(1)	107.4(4)	C(19)–B–C(37)	111.1(7)
C(46)–P–C(7)	113.9(4)	C(25)–B–C(31)	110.6(7)
C(46)–P–C(13)	108.3(4)	C(25)–B–C(37)	107.6(7)
C(1)–P–C(7)	108.9(3)	C(31)–B–C(37)	110.3(7)
C(1)–P–C(13)	109.4(3)		
C(7)–P–C(13)	108.7(3)		

TABLE IV. Some Least-squares Planes in the Form Px + Qy + Rz = S in the Direct Space (deviations in Å of the Atoms from the planes are given in brackets).

	P	Q	R	S
Plane (1)				
C(43) C(44) C(45) C(46) C(47)	6.0516	–5.9861	11.0820	4.7075
[C(43) –0.012, C(44) 0.012, C(45) –0.008, C(46) 0.001, C(47) 0.007, Rh –1.923, P 0.232]				
Plane (2)				
C(48) C(49) C(52) C(53)	7.722	–5.6267	10.6679	1.3662
[C(48) –0.087, C(49) 0.087, C(52) –0.083, C(53) 0.083, C(50) –0.826, C(51) –1.220, C(54) –1.065, C(55) –1.065, Rh 1.513]				
Plane (3)				
C(48, 49), C(52, 53)	5.0183	10.7621	1.0940	3.8270
Re (Rh 0.035)				
		Angles (°)		
	1.2 7.4	1–3 89.7	2–3	85.8

cm^{-1}. All diffraction experiments were performed on a Philips PW 1100 four-circle diffractometer by using graphite-monochromatized MoK$_\alpha$ radiation. Twenty reflections between 6 and 12° in θ were located by a random search procedure and subsequently centered. These reflections were used as basis for indexing. The cell constants and the orientation matrix obtained were refined by a least-squares fit. Intensities were collected at room temperature in the range $2° \leqslant \theta \leqslant 25°$. A θ–2θ scan mode was used with a scan speed of 0.03° sec^{-1}, a scan width of 1.20° and a 10 sec background count at each end of the scan.

No reflections were sufficiently intense to require the insertion of attenuators into the beam. After every 60 reflections approximately 2 standards were measured to check the crystal stability and electronics.

Of the 7480 reflections thus considered, 2790 had a net intensity greater than $3\sigma(I)$ (σ is the standard error based on count statistics) and these were used in the structure determination and refinement.

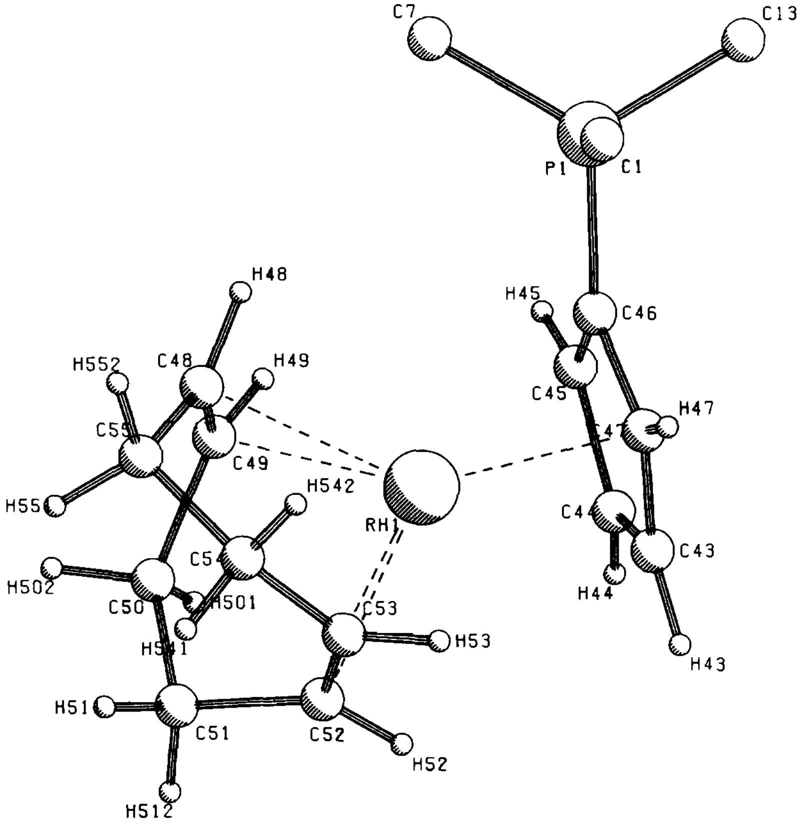

Fig. 1. View of the cation down *b*.

The data were corrected for Lorentz and polarization effects.

Structure Determination

The rhodium atom was located from a Patterson synthesis, and all the remaining non-hydrogen atoms by successive electron-density difference synthesis. The structure was refined by full matrix least-squares analysis. The phenyl rings were refined as rigid groups (D_{6h} symmetry) (C–C 1.395 Å, C–C–C 120°) and the hydrogen atoms were positioned in accordance with the expected geometry (C–H 0.95 Å) with a unique thermal parameter of 6 $Å^2$. Each C atom was assigned an individual isotropic thermal parameter. All atoms except those at fixed geometry were refined anisotropically. The final R and Rw values were respectively 0.0758 and 0.0760. The weighting scheme was chosen to give average values of $w\Delta^2$ for groups of reflections independent of the values of F_o and $\sin\theta/\lambda$ was $w = 2.118/(\sigma^2(F) + 0.001408\ F^2)$.

The SHELX system [8] of computer programs was used. Atomic scattering factors were taken from ref. [9], as were the corrections for the real and imaginary parts of the anomalous dispersion for rhodium.

The final difference Fourier showed no significant peaks. Final positional and isotropic thermal parameters are given in Table I, and the anisotropic ones in Table II. Bond distances and angles are in Table III and least-squares planes in Table IV.

Observed and calculated structure factors are available on request from the authors.

Results and Discussion

The stereochemistry of the cation is shown in Figs. 1 and 2 together with the atomic numbering scheme. Figure 1 is a projection of the cation down *b* and Fig. 2 is a view down *c*. (The phenyl rings are omitted for clarity). In Fig. 3 is reported the unit cell content viewed down *c*.

The rhodium atom is bonded to the substituted cyclopentadienyl ring (Rh–C 2.22(1)–2.29(1), average 2.26 Å) and to the two double bonds in cycloocta-1,5-diene (Rh–C 2.10, 2.04 Å). The planar Cp ring is almost parallel to the plane containing the two double bonds in the diene (angle of intersection 7.4°). The atoms of the diene group are however significantly removed from planarity with devia-

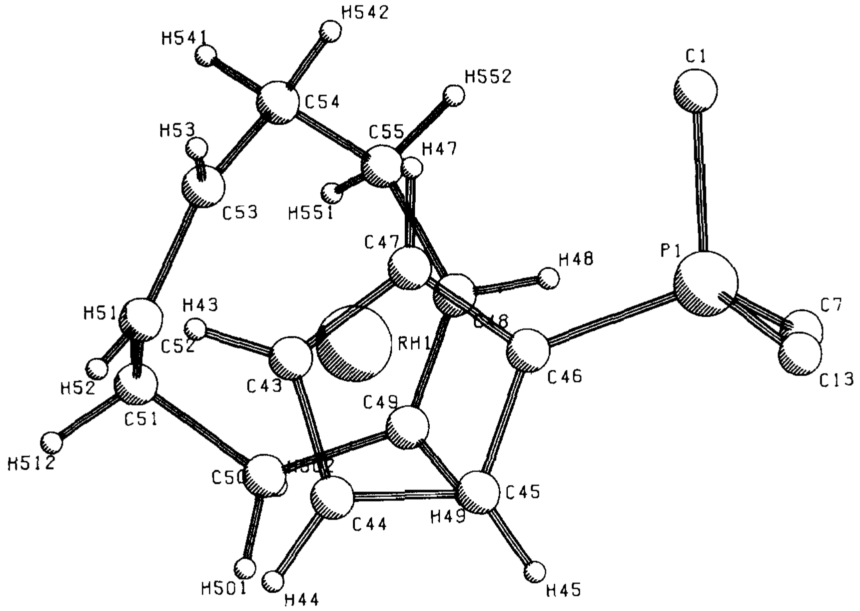

Fig. 2. View of the cation down *c*.

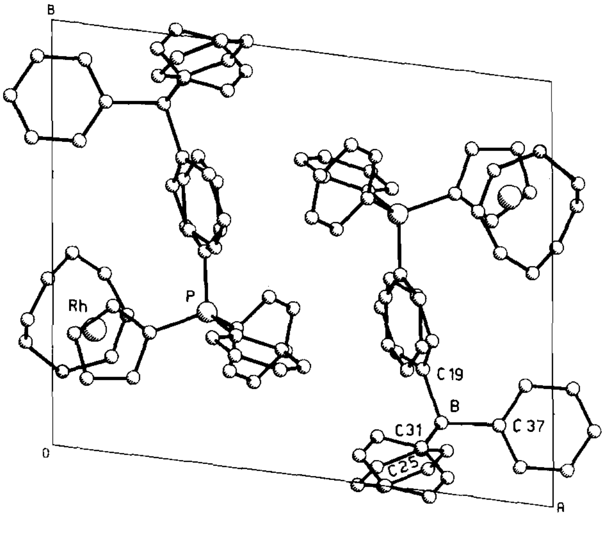

Fig. 3. Unit cell content viewed down *c*.

tions from the best mean plane ranging from –0.087 to 0.083 Å).

The rhodium atom, the midpoints of C(48,49), C(52,53), and the centroid of the Cp ligand (Rh–ring center 1.92 Å) define a plane from which the metal atom deviates by only 0.035 Å. The ligands are essentially trigonally disposed around the rhodium. The phosphorus atom is not coplanar to the Cp ring, being 0.232 Å apart. There are some significant differences amount the Rh–CCp bond distances.

The longest bonds are those from the metal to C(44) and C(45) [2.29(1) Å] while the shortest bonds are to C(43) and C(47) [2.22(1) and 2.23(1) Å]. The Rh–C(46) distance [2.27(1) Å] is closer in magnitude to Rh–C(44) and Rh–C(45).

A similar variation in Rh–C bond lengths was noted in the complex cycloocta-1,5-diene(η-methoxycarbonyl-cyclopentadienyl)rhodium(I) [10] in which the distances vary between 2.226(5) and 2.303(8) Å, in close agreement with the present structural study. Differences in the Cp ring C–C distances are comparable with the standard deviations and little significance can be placed on them.

The Rh–C bond lengths to the cycloocta-1,5-diene (cod) are also different, being 2.28(2) and 2.13(1) Å for Rh–C(48) and Rh–C(49) and 2.18(2) and 2.13(1) Å for Rh–C(52) and Rh–C(53) (average 2.18 Å). These values are longer than for corresponding bond lengths in other complexes of Rh with this diene. Thus in $Rh(cod)(C_5H_4CO_2Me)$ [10] and Rh(cod)(acac) [11] the Rh–C(cod) distances are 2.115 and 2.103 (acac = pentane-2,4-dionate). In this case the Rh–C(cod) bonds lengths are similar to those in complexes where the alkene is ethylene [2,167(2) in $Rh(Cp)(C_2H_4)(C_2F_4)$ [12] and 2.19(1) in $Rh(C_2H_4)(C_2F_4)(acac)$ [13]].

The cyclooctadiene ligand has a skewed conformation as observed in the free molecule [14]. When coordinated to the metal, as for example in Rh(cod)-(acac) [11], there is a significant difference between $C_{51}\cdots C_{55}$ and $C_{50}\cdots C_{54}$ distances across the ring: this is 0.68 Å in the free molecule [14], 0.43 Å in Rh(cod)(acac) [11], and 0.49 in the present compound (distances 3.05 and 3.54 Å respectively). On the basis of Tucker's [11] consideration that the difference between these two values decreases as the ligand becomes more strongly bonded to a metal we should expect short Rh–C(cod) bond distances. On the contrary we found Rh–C(cod) distances longer than expected. However there is a correlation between the Rh–CCp bonds and the Rh–C(cod) bonds. The longer Rh–C(48) (2.28 Å) bond is *trans* to the shorter Rh–C(43) (2.22 Å) (the angle C(43)–Rh–C(48) is 168°) and the shorter Rh–C(53) (2.13 Å) is opposite to Rh–C(45) (2.29 Å) (The angle C(53)–Rh–C(45) is 161°).

A close examination of the contact distances between the hydrogen of the diene and the rhodium atom shows the values Rh···H(48) 2.52, Rh···H(49) 2.28, Rh···H(52) 2.39, Rh···H(53) 2.32 Å. The presence of short Rh···H contacts could justify, in the present case, the lenghtening of the Rh···Cod bond distances better than the ligand symmetry.

The mean C=C length in cod is 1.34 Å, equal within experimental error to the equivalent lengths in the free molecule [14] (1.341 Å), in (cod)duroquinonenickel (1.325 Å) [15], and in (cod)Rh($C_6H_5CO_2Me$) [10] (1.388 Å). The Csp^2–Csp^3 lengths and the ring angles within the cod ligand are comparable with those reported in the free molecule and in other metal complexes of the ligand.

However it is interesting to notice the change in the cod conformation in the very similar compounds Rh(cod)($C_5H_5CO_2Me$) [10] and Rh(cod)(C_5H_4-$P(C_6H_5)_3$, being boat for the first and asymmetric skewed in the second as generally found for complexes containing the cod ligand.

The π bonded ylide ligand has geometry and bond lengths as found in (Ph_3P–C_5H_4)$Co(CO)_3$ cation [4].

The tetraphenylborate group has the usual tetrahedral geometry. There are no intermolecular contacts shorter than the sum of van der Waals atomic radii, in particular there is no interaction between the phenyl group reflecting an inefficient packing between cationic and anionic units.

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