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The Molecular Structure of μ-Carbonyl-di-μ-diphenylmethylene-bis(π-cyclopentadienylrhodium), $[(\pi\text{-}C_5H_5)_2Rh_2\{C(C_6H_5)_2\}_2(CO)]$

Hiroshi Ueda, Yasushi Kai, Noritake Yasuoka, and Nobutami Kasai*

Department of Applied Chemistry, Faculty of Engineering, Osaka University,
Yamadakami, Suita, Osaka 565

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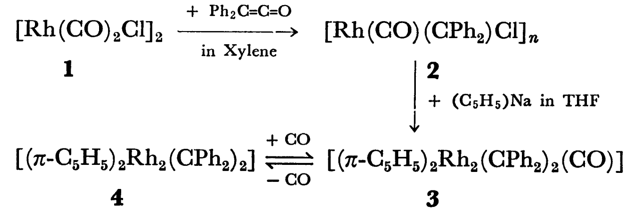
The molecular structure of $[(\pi\text{-}C_5H_5)_2Rh_2\{C(C_6H_5)_2\}_2(CO)]$ has been determined from three-dimensional X-ray diffraction data ($R=0.040$ for 2394 reflections). The crystal belongs to the monoclinic system, space group A2/a, with four formula units in a cell of dimensions: $a=19.967(5)$, $b=10.343(1)$, $c=16.526(4)$ Å, and $\beta=123.05(2)°$. The molecule has C_2 symmetry: the two-fold axis passes through the bridge carbonyl group, being perpendicular to the Rh–Rh bond (Rh—Rh=2.548(1) Å). The diphenylcarbene moieties bridge between rhodium atoms. The planar cyclopentadienyl rings lie 1.94 Å from the nearest rhodium atom and exihibit the usual π-bonded geometry.

In recent years many transition metal-carbene complexes have been reported[1–4)], in which the carbene ligands are stabilized by an adjacent oxygen or nitrogen atom. Recently, P. Hong and co-workers[5)] have prepared some stable diphenyl-carbene complexes, which have no hetero atom adjacent to the carbene ligand.

The reaction between $[Rh(CO)_2Cl]_2$ (**1**) and diphenyl-ketene gives an insoluble, brick red complex $[Rh(CO)\{C(C_6H_5)\}_2Cl]_n$ (**2**). The reaction of **2** with cyclopentadienyl sodium in THF gives air stable, green, needle-like crystals **3**. The complex **3** in solution slowly loses the bridging carbonyl on heating, and finally changes into **4**. The reaction from **3** to **4** is reversible.



$$\underset{\mathbf{1}}{[Rh(CO)_2Cl]_2} \xrightarrow[\text{in Xylene}]{+\ Ph_2C{=}C{=}O} \underset{\mathbf{2}}{[Rh(CO)(CPh_2)Cl]_n}$$

$$\downarrow +\ (C_5H_5)Na\ \text{in THF}$$

$$\underset{\mathbf{4}}{[(\pi\text{-}C_5H_5)_2Rh_2(CPh_2)_2]} \underset{-\,CO}{\overset{+\,CO}{\rightleftharpoons}} \underset{\mathbf{3}}{[(\pi\text{-}C_5H_5)_2Rh_2(CPh_2)_2(CO)]}$$

X-Ray molecular structures of **3** and **4** have been determined. This paper describes the crystal and molecular structure determination of **3**.

Experimental

The crystals used in this study were supplied by Professor N. Hagihara and co-workers. After preliminary X-ray work which showed that the crystal belongs to the monoclinic system, the unit-cell dimensions were determined by a least-squares fit using 2θ-values of 20 strong reflections measured on a G. E. single crystal orienter equipped on a Rigaku SG-2 goniometer.

Crystal Data. $C_{37}H_{30}ORh_2$, F. W. 696.5, $a=19.967(5)$, $b=10.343(1)$, $c=16.526(4)$ Å, $\beta=123.05(2)°$, $U=2860.0(9)$ Å^3, $D_m=1.60$ g cm^{-3}, $Z=4$, $D_c=1.62$ g cm^{-3}, $\mu(\text{Mo})=11.2$ cm^{-1}.

The systematic absences of reflections, hkl: $k+l=2n+1$ and $h0l$: $h=2n+1$, suggested the probable space group as A2/a (No. 16) or Aa (No. 9). Besides these diffraction aspects, the intensities of $h=2n+1$ layers were generally much weaker than those of $h=2n$ layers.

A single crystal with dimensions *ca.* $0.18\times0.21\times0.24$ mm was used to collect intensity data. Three-dimensional data were measured by means of the θ-2θ scan technique on a Rigaku automated, four-circle diffractometer using zirconium-filtered Mo $K\alpha$ radiation. The integrated intensity was determined by scanning over a peak at a rate of 4°min^{-1}, and subtracting the background by averaging the two values measured for 5 s at both ends of a scan. The scan width $\Delta(2\theta)$ of each reflection was computed by the equation: $\Delta(2\theta)=(2.0+0.70\tan\theta_c)°$, where θ_c is the calculated value of the Bragg angle for Mo $K\alpha_1(\lambda=0.70926$ Å). The intensities of three standard reflections were measured after every 50 reflections. There was no evidence of crystal decomposition. After reflections up to $\sin\theta/\lambda=0.481$ were measured, reflections with $h=2n$ in the range $0.47\leqq\sin\theta/\lambda\leqq0.66$ were also collected since the intensities of high angle reflections with $h=2n+1$ were too weak to be measured. A total of 2394 independent reflections was obtained. Lorentz and polarization corrections were made in the usual manner, but no absorption correction was applied.

Structure Solution and Refinement

The structure was solved by using a three-dimensional Patterson function. A clue for the solution of the structure was obtained from the characteristic intensity distribution of the reflections mentioned above, from which it could be assumed that the space group A2/a was preferable and the y parameter of the rhodium atom nearly equal to zero. This is consistent with the Patterson function. The first Fourier map phased by the rhodium atom was confusing owing to the pseudo-mirror at $y=0$. However, light atoms consisting of a phenyl ring and a carbonyl group could be located. The remaining non-hydrogen atoms were found from subsequent Fourier syntheses.

The structure was refined anisotropically by the block-diagonal least-squares procedure minimizing $\sum(|F_o|-k|F_c|)^2$. The HBLS V program was used.[6)] The hydrogen atoms, located by a difference Fourier map, were refined isotropically. The final R value is 0.040 for non-zero reflections. Atomic scattering factors used were those of Hanson, Herman, Lea and Skillman.[7)] The positional and thermal parameters are given in Table 1.**

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* To whom any correspondence should be addressed.

** The table of structure factors is kept as Document No. 7714 at the Chemical Society of Japan.

Table 1. Atomic positional and thermal parameters

Estimated standard deviations in parentheses. Anisotropic thermal parameters are expressed in the form $\exp\{-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl)\}$ and isotropic thermal parameters $\exp\{-B\sin^2\theta/\lambda^2\}$.

Atom	x	y	z	B or β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Rh	0.19361(2)	0.00744(5)	0.01464(3)	0.00222(1)	0.00631(4)	0.00379(2)	−0.00147(5)	0.00300(3)	−0.00044(7)
O	0.25000	−0.2523(6)	0.0	0.0055(4)	0.0037(6)	0.0099(6)	0.0	0.0038(8)	0.0
C(1)	0.3075(3)	0.0893(5)	0.1015(4)	0.0018(2)	0.0049(5)	0.0035(3)	−0.0006(5)	0.0028(4)	−0.0002(6)
C(2)	0.25000	−0.1409(8)	0.0	0.0023(3)	0.0064(8)	0.0048(5)	0.0	0.0020(7)	0.0
C(11)	0.3200(4)	0.2333(5)	0.1135(4)	0.0020(2)	0.0056(5)	0.0036(3)	−0.0009(5)	0.0022(4)	−0.0004(6)
C(12)	0.3947(4)	0.2822(6)	0.1448(5)	0.0027(2)	0.0065(6)	0.0058(4)	−0.0006(6)	0.0032(6)	0.0003(6)
C(13)	0.4116(4)	0.4123(7)	0.1605(6)	0.0034(3)	0.0092(8)	0.0074(5)	−0.0046(8)	0.0026(6)	−0.0006(10)
C(14)	0.3538(5)	0.4965(7)	0.1468(5)	0.0053(3)	0.0061(6)	0.0063(4)	−0.0027(10)	0.0032(6)	−0.0032(11)
C(15)	0.2810(4)	0.4513(7)	0.1197(5)	0.0046(3)	0.0072(6)	0.0055(4)	0.0034(8)	0.0041(6)	−0.0011(9)
C(16)	0.2632(4)	0.3204(6)	0.1029(5)	0.0027(2)	0.0063(6)	0.0047(4)	0.0001(6)	0.0026(5)	−0.0012(7)
C(21)	0.3531(4)	0.0222(6)	0.1969(4)	0.0020(2)	0.0069(7)	0.0038(3)	−0.0024(5)	0.0024(5)	−0.0005(6)
C(22)	0.3637(4)	0.0834(7)	0.2788(6)	0.0032(3)	0.0113(8)	0.0044(4)	−0.0003(7)	0.0034(6)	−0.0016(9)
C(23)	0.4015(5)	0.0235(8)	0.3671(5)	0.0048(3)	0.0146(11)	0.0040(4)	−0.0019(9)	0.0038(6)	−0.0007(10)
C(24)	0.4316(5)	−0.0986(8)	0.3800(5)	0.0042(3)	0.0152(10)	0.0043(4)	−0.0052(10)	0.0015(6)	0.0064(11)
C(25)	0.4245(5)	−0.1608(7)	0.3022(5)	0.0044(3)	0.0088(8)	0.0055(4)	−0.0021(10)	0.0012(6)	0.0040(10)
C(26)	0.3852(4)	−0.1010(6)	0.2121(5)	0.0038(3)	0.0071(6)	0.0043(4)	−0.0018(7)	0.0024(5)	−0.0000(8)
C(31)	0.0911(5)	0.0931(9)	0.0189(6)	0.0037(3)	0.0163(11)	0.0089(6)	−0.0029(10)	0.0092(7)	−0.0036(13)
C(32)	0.1489(5)	0.0428(10)	0.1119(6)	0.0041(3)	0.0278(13)	0.0060(5)	−0.0074(7)	0.0074(7)	−0.0066(14)
C(33)	0.1533(6)	−0.0927(10)	0.1063(7)	0.0059(4)	0.0192(13)	0.0085(6)	−0.0029(12)	0.0096(9)	0.0064(15)
C(34)	0.1000(5)	−0.1318(9)	0.0098(7)	0.0054(4)	0.0146(11)	0.0104(7)	−0.0092(11)	0.0094(8)	−0.0026(14)
C(35)	0.0612(4)	−0.0180(10)	−0.0452(6)	0.0024(2)	0.0218(13)	0.0075(5)	−0.0066(10)	0.0065(6)	−0.0069(14)
H(12)	0.436(3)	0.225(5)	0.156(4)	0.1(10)					
H(13)	0.452(4)	0.431(7)	0.177(4)	3.6(18)					
H(14)	0.362(4)	0.577(6)	0.158(4)	1.8(14)					
H(15)	0.246(4)	0.504(6)	0.103(4)	1.4(12)					
H(16)	0.211(3)	0.287(5)	0.080(4)	0.8(11)					
H(22)	0.341(4)	0.168(7)	0.270(4)	2.5(16)					
H(23)	0.410(4)	0.075(7)	0.425(5)	2.8(17)					
H(24)	0.461(4)	−0.141(6)	0.446(4)	1.9(14)					
H(25)	0.449(4)	−0.242(7)	0.306(5)	3.4(18)					
H(26)	0.384(4)	−0.139(6)	0.166(4)	1.5(14)					
H(31)	0.079(3)	0.208(8)	0.001(5)	3.9(19)					
H(32)	0.179(5)	0.115(8)	0.169(6)	5.0(21)					
H(33)	0.186(5)	−0.161(8)	0.159(6)	4.6(21)					
H(34)	0.088(5)	−0.230(9)	−0.021(6)	5.5(23)					
H(35)	0.020(4)	−0.019(7)	−0.115(5)	3.7(18)					

Results and Discussion

The molecular structure and the numbering scheme of atoms are shown in Fig. 1. A stereoscopic drawing of the molecule is given in Fig. 2. Bond lengths and bond angles are listed in Table 2. Table 3 shows the equations of several least-squares planes.

The remarkable feature of the molecule is that it has C_2 symmetry; a crystallographic two fold axis passes through the bridging carbonyl groups, being perpendicular to the Rh–Rh bond. The diphenylmethylene moieties bridge between rhodium atoms instead of bonding to single rhodium atom. Each of the cyclopentadienyl group is π-bonded to a rhodium atom, and the two cyclopentadienyl rings are staggered about the Rh–Rh bond (Fig. 1).

The Rh–Rh single bond length 2.548(1) Å is significantly shorter than those in other π-cyclopentadienyl rhodium carbonyl complexes: 2.681(2) Å in $[(\pi\text{-}C_5H_5)_2Rh_2(CO)_3]$,[9] 2.62 Å in $[(\pi\text{-}C_5H_5)_3Rh_3(CO)_3]$,[10] 2.776(1) Å in $Rh_6(CO)_{16}$[11] and 2.690 Å in rhodium metal.[12]

The cyclopentadienyl rings are planar: the maximum deviation from the plane is ±0.001 Å. The C–C bond lengths and C–C–C bond angles are 1.411(16)—1.453 (14) (av. 1.425) Å, and 105.5(9)—109.7(9) (av. 108.0)°, respectively. Each ring makes an angle of 83° to the Rh–Rh bond and lies 1.94 Å away from the rhodium atom. The average Rh–C(C_5H_5) distance is 2.290(12) Å, which is comparable to the values of 2.24 Å in $[(\pi\text{-}C_5H_5)_3Rh_3(CO)_3]$, 2.26 Å in $[(\pi\text{-}C_5H_5)_2Rh_2(CO)_3]$, and 2.25 Å in $[(\pi\text{-}C_5H_5)Rh(CO)(C_2F_5)I]$.[13]

The Rh–C(1) bond lengths are 2.094(6) and 2.088 (6) (av. 2.091) Å, which are close to the Rh(III)–C-

TABLE 2. BOND LENGTHS AND BOND ANGLES

Bond lengths [l/Å]

Rh-Rh′	2.548(1)	Rh-C(2)	1.995(9)	C(26)-C(21)	1.386(10)	C(31)-C(32)	1.421(15)
Rh-C(1)	2.094(6)	Rh′-C(1)	2.088(6)	C(32)-C(33)	1.412(15)	C(33)-C(34)	1.411(16)
Rh-C(31)	2.266(10)	Rh-C(32)	2.271(11)	C(34)-C(35)	1.430(10)	C(35)-C(31)	1.453(14)
Rh-C(33)	2.312(11)	Rh-C(34)	2.325(11)				
Rh-C(35)	2.276(10)	C(1)-C(1)	2.851(12)	C(12)-H(12)	0.95(7)	C(13)-H(13)	0.72(9)
C(1)-C(11)	1.505(9)	C(1)-C(21)	1.493(9)	C(14)-H(14)	0.85(8)	C(15)-H(15)	0.83(7)
C(2)-O	1.152(10)	C(11)-C(12)	1.381(10)	C(16)-H(16)	0.96(7)	C(22)-H(22)	0.96(8)
C(12)-C(13)	1.377(12)	C(13)-C(14)	1.363(13)	C(23)-H(23)	1.02(9)	C(24)-H(24)	1.01(8)
C(14)-C(15)	1.349(13)	C(15)-C(16)	1.388(11)	C(25)-H(25)	0.96(9)	C(26)-H(26)	0.85(8)
C(16)-C(11)	1.384(10)	C(21)-C(22)	1.402(10)	C(31)-H(31)	1.22(9)	C(32)-H(32)	1.08(10)
C(22)-C(23)	1.372(12)	C(23)-C(24)	1.364(13)	C(33)-H(33)	1.03(10)	C(34)-H(34)	1.10(11)
C(24)-C(25)	1.375(13)	C(25)-C(26)	1.393(12)	C(35)-H(35)	0.99(9)		

Bond angles [φ/°]

Rh′-Rh-C(2)	50.3(2)	Rh′-Rh-C(1)	52.4(2)	H(26)-C(26)-C(21)	118(5)
Rh-Rh′-C(1)	52.6(2)	C(2)-Rh-C(1)	85.5(3)	H(31)-C(31)-C(32)	124(4)
C(2)-Rh′-C(1)	85.6(3)	C(1)-Rh-C(1)′	86.0(2)	H(32)-C(32)-C(33)	136(5)
Rh-C(2)-Rh′	79.4(3)	Rh-C(2)-O	140.3(7)	H(33)-C(33)-C(34)	120(5)
Rh-C(1)-Rh′	75.1(2)	Rh-C(1)-C(11)	122.1(4)	H(34)-C(34)-C(35)	124(5)
Rh′-C(1)-C(11)	116.1(4)	Rh-C(1)-C(21)	110.2(4)	H(35)-C(35)-C(31)	128(5)
Rh-C(1)-C(21)	118.4(4)	C(11)-C(1)-C(21)	111.1(5)	C(11)-C(12)-H(12)	120(4)
C(16)-C(11)-C(12)	116.9(6)	C(11)-C(12)-C(13)	122.2(7)	C(12)-C(13)-H(13)	116(7)
C(12)-C(13)-C(14)	119.7(8)	C(13)-C(14)-C(15)	119.6(9)	C(13)-C(14)-H(14)	124(5)
C(14)-C(15)-C(16)	121.2(8)	C(15)-C(16)-C(11)	120.4(7)	C(14)-C(15)-H(15)	118(5)
C(26)-C(21)-C(22)	115.4(6)	C(21)-C(22)-C(23)	121.9(7)	C(15)-C(16)-H(16)	123(4)
C(22)-C(23)-C(24)	121.6(8)	C(23)-C(24)-C(25)	118.4(8)	C(21)-C(22)-H(22)	117(5)
C(24)-C(25)-C(26)	120.2(8)	C(25)-C(26)-C(21)	122.5(7)	C(22)-C(23)-H(23)	118(5)
C(35)-C(31)-C(32)	105.5(9)	C(31)-C(32)-C(33)	109.7(9)	C(23)-C(24)-H(24)	121(4)
C(32)-C(33)-C(34)	108.8(10)	C(33)-C(34)-C(35)	107.2(9)	C(24)-C(25)-H(25)	124(5)
C(34)-C(35)-C(31)	108.8(9)			C(25)-C(26)-H(26)	119(5)
		H(16)-C(16)-C(11)	117(4)	C(35)-C(31)-H(31)	131(4)
H(12)-C(12)-C(13)	118(4)	H(22)-C(22)-C(23)	121(5)	C(31)-C(32)-H(32)	115(5)
H(13)-C(13)-C(14)	124(7)	H(23)-C(23)-C(24)	120(5)	C(32)-C(33)-H(33)	131(5)
H(14)-C(14)-C(15)	117(5)	H(24)-C(24)-C(25)	120(4)	C(33)-C(34)-H(34)	129(5)
H(15)-C(15)-C(16)	120(5)	H(25)-C(25)-C(26)	115(5)	C(34)-C(35)-H(35)	123(5)

(sp^3) distances of 2.08(3) in $[(\pi\text{-}C_5H_5)Rh(CO)(C_2F_5)I]$ and 2.08 Å in $[Rh_2I_2(SMe_2)_3Me_4]$.[14]

In the diphenylmethylene moiety, bond angles around the C(1) atom deviate from the tetrahedral angle: Rh–C(1)–Rh′ 75.1(2), Rh–C(1)–C(11) 122.1(4), Rh–C(1)–C(21) 110.2(4), Rh′–C(1)–C(11) 116.1(4), Rh′–C(1)–C(21) 118.4(4), C(11)–C(1)–C(21) 111.1(5)°. Bond lengths and bond angles in phenyl rings have normal values [C–C(av.) = 1.38 Å and C–C–C(av.) = 120.0°]. The two phenyl rings are roughly perpendicular to each other, the dihedral angle being 85.3°.

The Rh–C(2) bond length 1.995(9) Å is shorter than that of Rh–C(1). However, this distance is close to the values of 2.01 Å in $[(\pi\text{-}C_5H_5)_2Rh_2(CO)_3]$ and 2.00 Å in $[(\pi\text{-}C_5H_5)_3Rh_3(CO)_3]$. The bridging Rh–C(2)–Rh′ angle is 79.4(3)°, which is smaller than the corresponding angles of 84.0° in $[Rh_2(C_5H_5)_2(CO)_3]$, 81.9° in $[(\pi\text{-}C_5H_5)_3(CO)_3]$ and 82° in $[\{(PPh_3)_2Rh(CO)\}_2,2CH_2Cl_2]$.[15] The C–O length of the bridged carbonyl group, C(2)–O = 1.152(10) Å, is comparable to the values of 1.15(4) and 1.18(4) Å in $[(\pi\text{-}C_5H_5)_3Rh_3(CO)_3]$ and 1.17 Å in $[\{(PPh_3)Rh(CO)\}_2,2CH_2Cl_2]$.

The overall molecular geometry of the present complex, except for the bridging carbonyl group, is similar to that of the diamagnetic complex $[(C_6H_5)_2PCo(C_5H_5)]_2$.[16] The molecular structure of the cobalt complex was described by the formation of a so-called "bent" metal-metal bond. However, this is not the case for the present complex because the bridging carbonyl group prevents the formation of the "bent" metal-metal bond. The $(CPh_2)_2$ bridging system is not planar: the dihedral angle between the two planes formed by Rh, Rh′, and C(1) and Rh, Rh′, and C(1)′ being decreased from 180 to 121° (Fig. 1(b)). The intra- and inter-molecular atomic contacts indicate that the magnitude of twisting of the phenyl rings is primarily determined by non-bonding intra-molecular interactions. The dihedral angles formed by the plane defined by C(1), C(1)′ and C(2) with phenyl rings 1 and 2 are 61.1, 31.8°, respectively. Because of the short C(1)···

TABLE 3. LEAST-SQUARES PLANES AND ATOM DEVIATIONS

Plane equations are of the form $AX+BY+CZ+D=0$, where X, Y, Z and D are measured in Å units: $X=ax+cz\cos\beta$, $Y=by$ and $Z=cz\sin\beta$.

(a) Plane through Rh, C(2) and Rh′
$-0.1592X-0.9873Z+0.7945=0$
C(1)* 1.426 C(1)′* −1.426

(b) Plane through Rh, C(1)′ and Rh′
$-0.0813X+0.8598Y-0.5041Z+0.3395=0$

(b′) Plane through Rh, C(1) and Rh′
$-0.0813X-0.8598Y-0.5041Z+0.4719=0$

(c) Plane through C(1), C(1)′ and C(2)
$-0.9867X+0.1627Z+4.9253=0$

(d) Phenyl ring 1 (C(11)-C(16))
$-0.2694X-0.1258Y+0.9548Z+0.2350=0$
C(11) −0.014 C(13) −0.003 C(15) 0.009
C(12) 0.012 C(14) −0.016 C(16) 0.014

(d′) Phenyl ring 1′ (C(11)′-C(16)′)
$-0.2664X+0.1236Y+0.9559Z+2.4525=0$
C(11)′ 0.018 C(13)′ −0.002 C(15)′ −0.008
C(12)′ −0.013 C(14)′ 0.012 C(16)′ −0.008

(e) Phenyl ring 2 (C(21)-C(26))
$-0.8973X-0.3838Y-0.2182Z+5.4261=0$
C(21) 0.009 C(23) −0.001 C(25) −0.010
C(22) −0.009 C(24) 0.010 C(26) 0.000

(e′) Phenyl ring 2′ (C(21)′-C(26)′)
$-0.8973X+0.3838Y-0.2181Z+3.5325=0$
C(21)′ −0.009 C(23)′ 0.000 C(25)′ 0.010
C(22)′ 0.009 C(24)′ −0.010 C(26)′ −0.000

(f) Cyclopentadienyl ring (C(31)-C(35))
$0.9771X-0.1198Y+0.1757Z+1.6801=0$
C(31) −0.000 C(33) −0.001 C(35) −0.001
C(32) 0.001 C(34) 0.001 Rh* −1.942

(f′) Cyclopentadienyl ring (C(31)′-C(35)′)
$-0.9771X+0.1198Y+0.1757Z+8.0751=0$
C(31)′ 0.000 C(33)′ 0.001 C(35)′ 0.001
C(32)′ −0.001 C(34)′ −0.001 Rh′* 1.942

* Not included in the least-squares plane calculation.

DIHEDRAL ANGLES [φ/°]

Between planes	Dihedral angles
a, b	120.7
b, b′	118.6
c, d	61.1
c, e	31.8
d, d′	14.3
d, e	85.3
d, e′	90.8
e, f	45.1
f, f′	13.8

ANGLE BETWEEN INTERATOMIC VECTOR AND PLANE [φ/°]

Rh-Rh′ bond······C_5H_5 plane	83.1

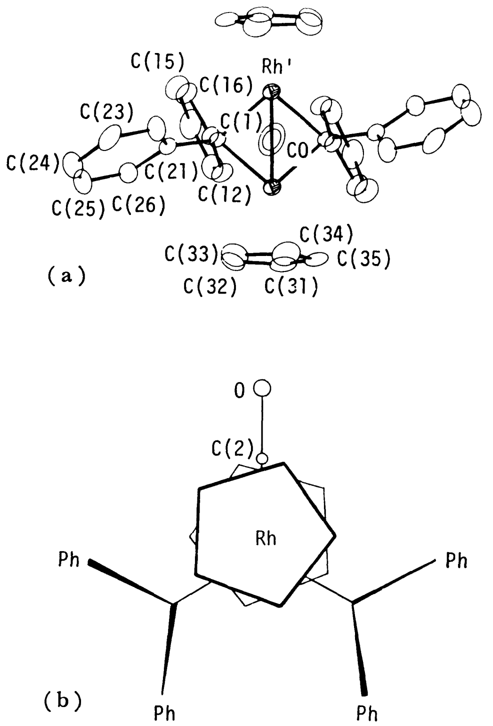

Fig. 1. The molecular structure of $[(\pi\text{-}C_6H_5)_2Rh_2(CO)(CPh_2)_2]$ and the numbering scheme of atoms.
(a) Projected along the C–O bond.
(b) Projected along the Rh-Rh bond. Rh atoms are omitted.

TABLE 4. SHORT INTERMOLECULAR ATOMIC CONTACTS LESS THAN 3.1 Å [l/Å]

H(14)···O^a	2.92(8)	H(15)···O^a	3.07(7)
H(26)···H(14)^b	2.97(11)	H(22)···H(33)^c	2.34(13)
H(15)···H(23)^c	2.96(11)	H(23)···H(34)^c	2.56(15)
H(24)···H(31)^d	2.14(12)		

key a x, $1+y$, z; b x, $-1+y$, z; c $0.5-x$, $0.5+y$, $0.5-z$; d $0.5-x$, $-0.5+y$, $0.5+z$.

C(1)′ distance of 2.851(12) Å, the phenyl rings 1 and 1′ related by the crystallographic two-fold axis are not parallel to each other: C(11)···C(11)′=3.232(13) Å, C(12)···C(16)′=3.578(11) Å and C(14)···C(15)′=3.733(13) Å, and the dihedral angle between the phenyl rings 1 and 1′ is 14.3°.

A stereoscopic drawing of the crystal structure is shown in Fig. 3. Short intermolecular atomic contacts (less than 3.1 Å) are given in Table 4.

Computations throughout the present study were carried out on a NEAC 2200-700 computer at Osaka University.

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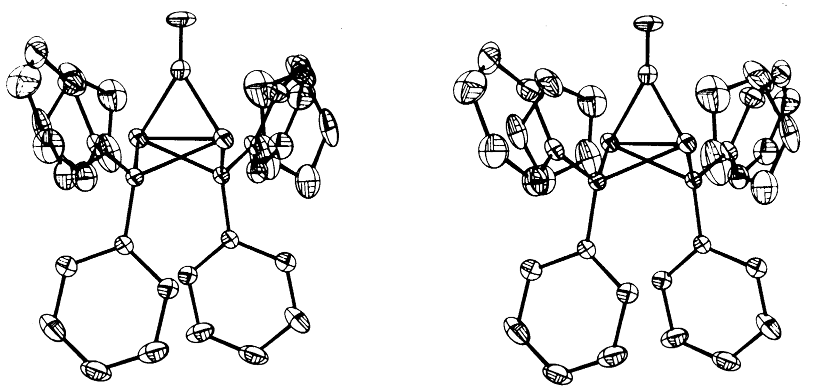

Fig. 2. A stereoscopic ORTEP drawing[8] of the molecule

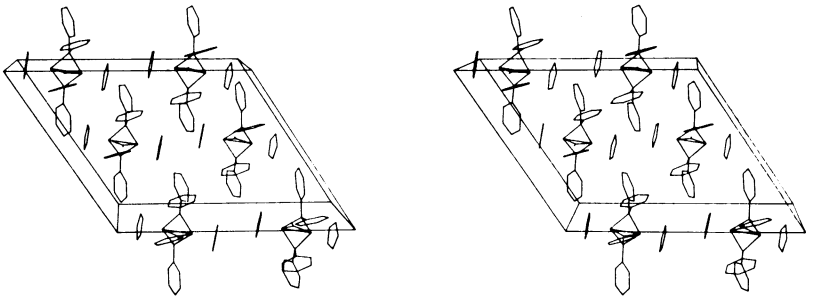

Fig. 3. A stereoscopic drawing[8] of the crystal structure projected along the b axis.

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