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Pentakis(methoxycarbonyl)cyclopentadiene chemistry

XI *. Preparation and characterisation of rhodium complexes containing ligands derived from 1,2,3-tris(methoxycarbonyl)cyclopentadiene: X-Ray crystal structure of Rh{ η^4-$C_5H_3(CO_2Me)_3$-1,4,5}{ η^5-$C_5H_2(CO_2Me)_3$-1,2,3}

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Abstract

Reactions between $Tl[C_5(CO_2Me)_5]$ and $\{RhCl(CO)_2\}_2$ afford Rh{η^4-$C_5H_3(CO_2Me)_3$-1,4,5}{η^5-$C_5H_2(CO_2Me)_3$}, fully characterised by an X-ray crystal structure determination, which is related to the previously described rhodicinium cation $[Rh\{\eta^5\text{-}C_5H_2(CO_2Me)_3\}_2]^+$ by formal addition of hydride to one of the C_5 rings. Full details of the preparation of the $[C_5(CO_2Me)_5]^-$ salt of the latter cation are also given. Crystal data: monoclinic, space group $P2_1/a$, a 19.543(7), b 13.52(1), c 18.789(7) Å, β 105.10(3)°, U 4794(5) Å^3, $Z = 8$, $N = 8342$, $N_o(I > 3\sigma(I)) = 3053$, $R = 0.090$, $R' = 0.092$.

Introduction

We have recently prepared a series of rhodium(I) and rhodium(III) complexes containing the pentakis(methoxycarbonyl)cyclopentadienyl group, $C_5(CO_2Me)_5$ [1]. In these, the latter group was variously present as the η^5, *C*-bonded ligand found in $Rh(\eta^4\text{-}C_8H_{12})\{\eta^5\text{-}C_5(CO_2Me)_5\}$ (**1**), as the η^2, *O*-bonded ligand found in

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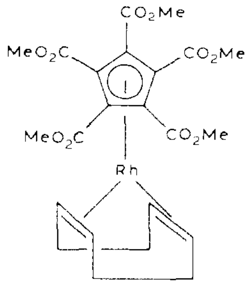
* For Part X, see Ref. 10.



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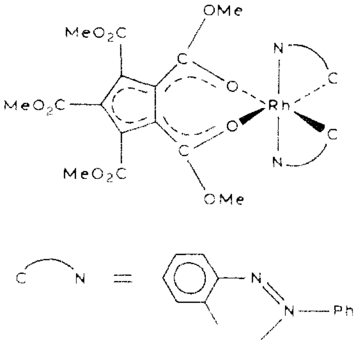

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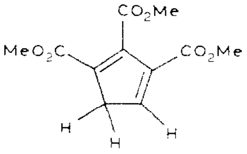

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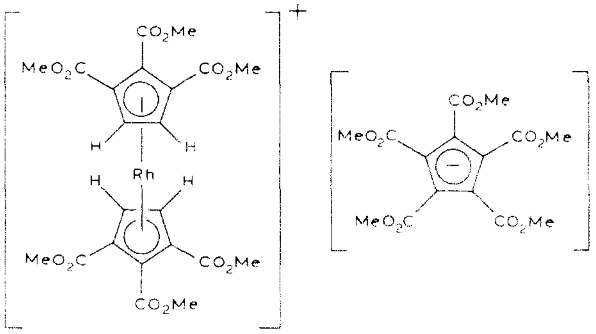

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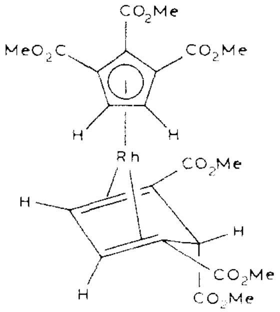


(5)

Rh(azb)$_2${$C_5(CO_2Me)_5$} (**2**), or as the very stable anion, $[C_5(CO_2Me)_5]^-$, in salts such as [Rh(dppe)(η^4-C_8H_{12})][$C_5(CO_2Me)_5$] and [Rh(CO)(NCMe)(PPh_3)$_2$][C_5-$(CO_2Me)_5$]. We now describe some related rhodium chemistry, which has afforded complexes containing ligands derived from 1,2,3,-tris(methoxycarbonyl)cyclopentadiene, $C_5H_3(CO_2Me)_3$ (**3**). Part of this work has been described in a preliminary report [2].

Results and discussion

The reaction between $Rh_2(OAc)_4$ and $HC_5(CO_2Me)_5$ gave red needles of a complex (**4**) shown by conductivity measurements to behave as a 1/1 electrolyte in polar solvents such as water or acetone. Microanalytical figures and an osmometric molecular weight of 1100 ± 10% in chloroform, suggested the presence of both ionic and covalently-bound C_5 fragments. A single-crystal X-ray diffraction study [2] showed that **4** has the illustrated structure, $[Rh\{\eta^5\text{-}C_5H_2(CO_2Me)_3\text{-}1,2,3\}_2][C_5(CO_2Me)_5]$, in which the cations and anions stack along the long axis of the needle crystals. The ^1H NMR spectrum contains three resonances at δ 3.75, 3.89 and 6.30, of relative intensities 18/15/4, which are assigned to accidentally equivalent CO_2Me groups of the $C_5H_2(CO_2Me)_3$ ligand, the $C_5(CO_2Me)_5$ anion, and the ring protons, respectively. The ^{13}C NMR spectrum is more informative, containing three inequivalent OMe resonances between δ 51.4 and 54.6 and three carbonyl resonances between δ 159.7 and 176.6; the ring carbons of the $C_5(CO_2Me)_5$ group resonate at δ 117.45, while those of the other η^5 ligands are found between δ 91.3 and 105.0.

The reaction between $Tl[C_5(CO_2Me)_5]$ and $\{RhCl(CO)_2\}_2$ was carried out in methanol. After several days at reflux point, TLC showed the presence of several products, of which only one has been isolated in a pure state. This compound (**5**) formed orange crystals, the mass spectrum of which contained the highest mass ion at *m/z* 582. In the ^1H NMR spectrum, seven singlet resonances of relative intensities 2/2/1/3/6/6/3 were present. These observations taken with the finding that complex **4** contained two $C_5H_2(CO_2Me)_3$ ligands, suggested that **5** also contained similar groups. Both the analytical results and the ion at *m/z* 582 were consistent with the formula $RhH\{C_5H_2(CO_2Me)_3\}_2$; no high-field ^1H resonance was found, and a single-crystal X-ray diffraction study was carried out to confirm the suggested diene-dienyl structure and to determine the precise CO_2Me substitution pattern of **5**.

As can be seen from Fig. 1, which shows a plot of one of the two independent molecules found in the unit cell, complex **5** is indeed another example of an 18e $Rh(\eta^4\text{-diene})(\eta^5\text{-dienyl})$ complex. The rhodium is attached to an $\eta^5\text{-}C_5H_2(CO_2Me)_3\text{-}1,2,3$ group and to the 1,3-diene system of the related 1,4,5-tris(methoxycarbonyl)-cyclopenta-1,3-diene ligand. The ^1H NMR spectrum can be interpreted on the basis of this structure and comparing it with that of **4**; the singlet at δ 4.19 is assigned to the *endo* proton on C(5), while the equivalent pairs of protons on the η^4 and η^5 ligands resonate at δ 5.64 and 5.98, respectively. Assignment of the two sets of CO_2Me resonances is not straightforward, apart from the obvious distinction between the single central and two outer CO_2Me groups on each ligand.

The Rh–C(cp) distances (2.24 Å) are comparable with those found in $Rh(\eta^4\text{-}C_8H_{12})\{\eta^5\text{-}C_5(CO_2Me)_5\}$ (2.28 Å) [1], but considerably longer than those in **4**, as would be expected when comparing neutral and cationic systems. The Rh–C(diene) separations (2.14 Å) are 0.1 Å shorter, and are similar to those found in $Rh(\eta^4\text{-}C_8H_{12})\{\eta^5\text{-}C_5(CO_2Me)_5\}$ (2.13 Å) [1]. The bulky CO_2Me group attached to C(2) of the diene is *exo*, while in the cp ligand, the middle substituent is twisted about its attachment similarly to ease steric strain.

In both cases, the isolation of **4** and **5** only after prolonged reaction times has precluded any studies designed to elucidate the manner in which the two CO_2Me groups are replaced by H. We have noted previously that model studies suggest that steric interactions between the CO_2Me groups of complexes $M\{\eta^5\text{-}C_5(CO_2Me)_5\}_2$ having metallocene 'sandwich' structures, would be severe as a result of the impossibility of all CO_2Me groups being able to adopt a configuration close to coplanarity with the C_5 ring. Inspection of the structure of **4** shows that the

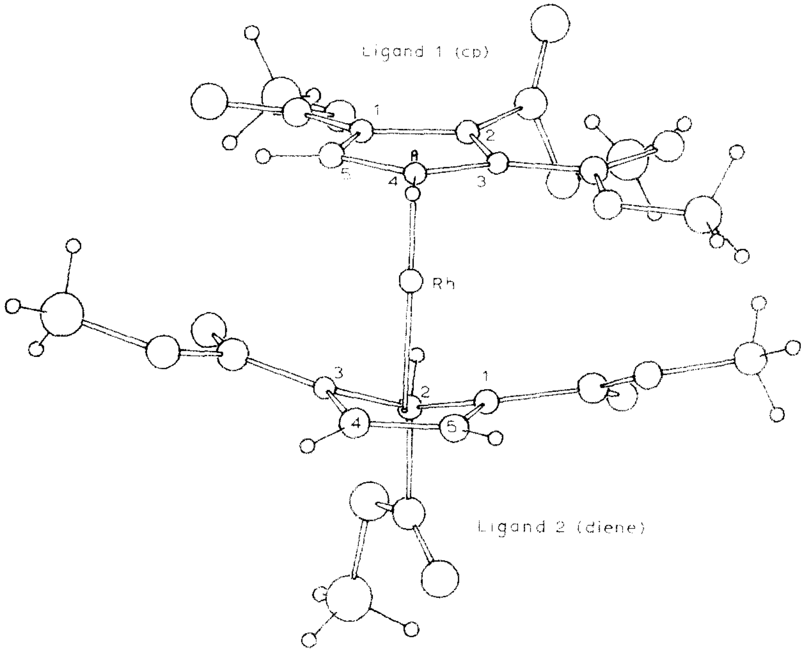
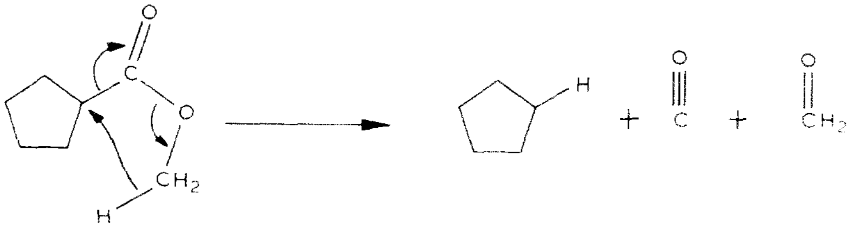


Fig. 1. A projection of molecule A of **5**, showing 20% thermal envelopes for the non-hydrogen atoms, with labelling, and hydrogen atoms of arbitrary radius 0.1 Å. (A similar projection of molecule B is indistinguishable.)

sterically least demanding conformation of the two rings is adopted, such that the CO_2Me groups of one ring lie above the H atoms of the second. No such constraint applies to **5**, since the central CO_2Me group on the η^5-ring, whose plane has a dihedral of ca. 80° with the C_5 ring plane, lies above and to one side of the proton attached to C(2) of the η^4-ring, i.e. the carbon furthest away (2.65 Å) from the metal, and thus is in the most open position. In consequence, the two CH–CH portions of the rings are approximately staggered but overlapping with respect to one another.

The steric interactions of CO_2Me groups on separate η^5-rings may provide the driving force for the reaction which results in the replacement of two of these groups on each ring with H. The reaction corresponds to a net elimination of "$C_2H_2O_2$" from each group, and we have observed similar processes in the mass spectra of $Ru(\eta\text{-}C_5H_5)\{\eta^5\text{-}C_5(CO_2Me)_5\}$ [3], where a stepwise conversion of the molecular ion of this complex to the molecular ion of ruthenocene, $[Ru(C_5H_5)_2]^+$, occurs. In that case, one can propose a cyclic elimination of CO and CH_2O:



Alternatively, abstraction of (2CO + 2H) may occur via the intermediacy of rhodium hydrido-carbonyl complexes, in a reversal of the rhodium-catalysed homologation reactions used in syn-gas chemistry.

Complexes **4** and **5** are related by hydride transfer reactions, as found previously for $[Rh(\eta\text{-}C_5H_5)_2]^+$ and nucleophiles such as Ph^- [4].

Conclusion

The formation and structures of two rhodium complexes containing ligands derived from tris(methoxycarbonyl)cyclopentadiene, itself formed by an unusual elimination of "$C_2H_2O_2$" ($CO + CH_2O$?) from two of the CO_2Me groups of the initial $C_5(CO_2Me)_5$ reagent, have been described.

Experimental

General experimental conditions have been described previously in [1]. The compounds $HC_5(CO_2Me)_5$ and $Tl[C_5(CO_2Me)_5]$ [5], $Rh_2(OAc)_4$ [6] and $\{RhCl(CO)_2\}_2$ [7] were prepared by published procedures.

Reaction between $Rh_2(OAc)_2$ and $HC_5(CO_2Me)_5$

Rhodium(II) acetate (140 mg, 0.32 mmol) was added to a stirred solution of $HC_5(CO_2Me)_5$ (500 mg, 1.4 mmol) in MeOH (10 ml), whereupon the blue-green MeOH adduct of the rhodium complex formed immediately. The mixture was heated at the reflux point for 48 h, after which cooling to room temperature afforded red needles; filtration, washing with MeOH (2 × 10 ml), and recrystallisation (MeOH) gave pure $[Rh\{\eta^5\text{-}C_5H_2(CO_2Me)_3\}_2][C_5(CO_2Me)_5]$ (**4**) (150 mg, 10%), m.p. 245 °C. Found: C, 47.28; H, 3.91; O, 37.45; $C_{22}H_{22}O_{12}Rh^+ \cdot C_{15}H_{15}O_{10}^-$ calc: C, 47.44; H, 3.95; O, 37.61%. IR (nujol): ν(CO) at 1745s, 1710s, 1670s; other bands at 1415m, 1365m, 1353m, 1255vs, 1175m, 1140m, 993m, 795m, 780m, 775m cm^{-1}. ^1H NMR: δ ($CDCl_3$) 3.73 (s, 18H, $(CO_2Me)_3$), 3.89 (s, 15H, $(CO_2Me)_5$), 6.30 (d, J(HRh) 1 Hz, 4H, CH). ^{13}C NMR: δ ($CDCl_3$) 51.38, (s, OMe), 117.45 (s, ring C), 167.80 (s, CO) for $C_5(CO_2Me)_5$ anion; 54.05 (s, 1,3-$(OMe)_2$), 54.60 (s, 2-OMe), 91.52 (d, J(CRh) 7 Hz) and 94.44 (d, J(CRh) 7 Hz) (*C*H and 1,3-*C*(CO_2Me), not distinguished), 104.8 (d, J(CRh) 7 Hz, 2-*C*(CO_2Me)), 159.78 (d, J(CRh) 5 Hz, 1,3-$(CO_2Me)_2$), 176.55 (s, 2-CO_2Me) ppm.

Reaction between $\{RhCl(CO)_2\}_2$ and $Tl[C_5(CO_2Me)_5]$

To a stirred solution of $Tl[C_5(CO_2Me)_5]$ (400 mg, 0.72 mmol) in MeOH (40 ml) was added $\{RhCl(CO)_2\}_2$ (150 mg, 0.38 mmol), to give a yellow solution. The mixture was refluxed for approximately 130 h to give a grey-black precipitate and a red-brown solution. The precipitate was filtered off and washed with MeOH (5 ml). The combined filtrates were evaporated to dryness (rotary evaporator). Preparative TLC of a CH_2Cl_2 extract of the residue (silica gel; 3/2 acetone/light petroleum) separated the major product (R_f 0.88) from several minor unidentified components. Crystallisation (MeOH) gave orange crystals of $Rh\{\eta^4\text{-}C_5H_3(CO_2Me)_3\}\{\eta^5\text{-}C_5H_2(CO_2Me)_3\}$ (**5**) (74 mg, 18%), m.p. 134–136 °C. Found: C, 44.94; H, 4.03%; *M* (mass spectrometry) 582; $C_{22}H_{23}O_{12}Rh$ calc: C, 45.38; H, 3.98%; *M*, 582. IR (Nujol): ν(C=O) 1750s, 1742s, 1735s, 1725s, 1717s, 1713s, 1708s, 1702(sh), 1690(sh), 1678(sh); other bands at 1440s, 1426m, 1411m, 1407(sh), 1350m, 1340m, 1332m, 1312s, 1297m, 1280m, 1257s, 1246(sh), 1205s, 1185s, 1170m, 1162(sh), 1140s, 1128m, 1074s, 1037(sh), 1033m, 1028m, 1000m, 960m, 778m, 770m cm^{-1}. ^{1}H NMR: δ ($CDCl_3$) 5.98 (s, 2H, CH), 5.64 (s, 2H, CH), 4.19 (s, 1H, CH), 3.91 (s, 3H, OCH_3), 3.81 (s, 6H, OCH_3), 3.70 (s, 6H, OCH_3), 3.55 (s, 3H, OCH_3). FAB MS (*m/z*, assignment, relative intensity): 582, $[M]^+$, 4; 580, $[M-2H]^+$, 4; 551, $[M-OMe]^+$, 14; 508, $[M-CO_2-OCH_2]^+$, 6; 522 $[M-CO_2Me-H]^+$, 100; 464, $[M-2CO_2Me]^+$, 9; 289, –, 5; 231, –, 8.

Crystallography

$Rh\{\eta^4\text{-}C_5H_3(CO_2Me)_3\}\{\eta^5\text{-}C_5H_2(CO_2Me)_3\}$ (5)

The crystals, although well-formed, diffracted poorly, giving weak and diffuse reflections; independent data sets were measured on two crystals, but the second was not appreciably better than the first. A unique data set was measured at 295 K within the limit $2\theta_{max} = 50°$ using a Syntex $P2_1$ four-circle diffractometer with monochromatic Mo-K_α radiation source (λ 0.71069 Å) in conventional $2\theta/\theta$ scan

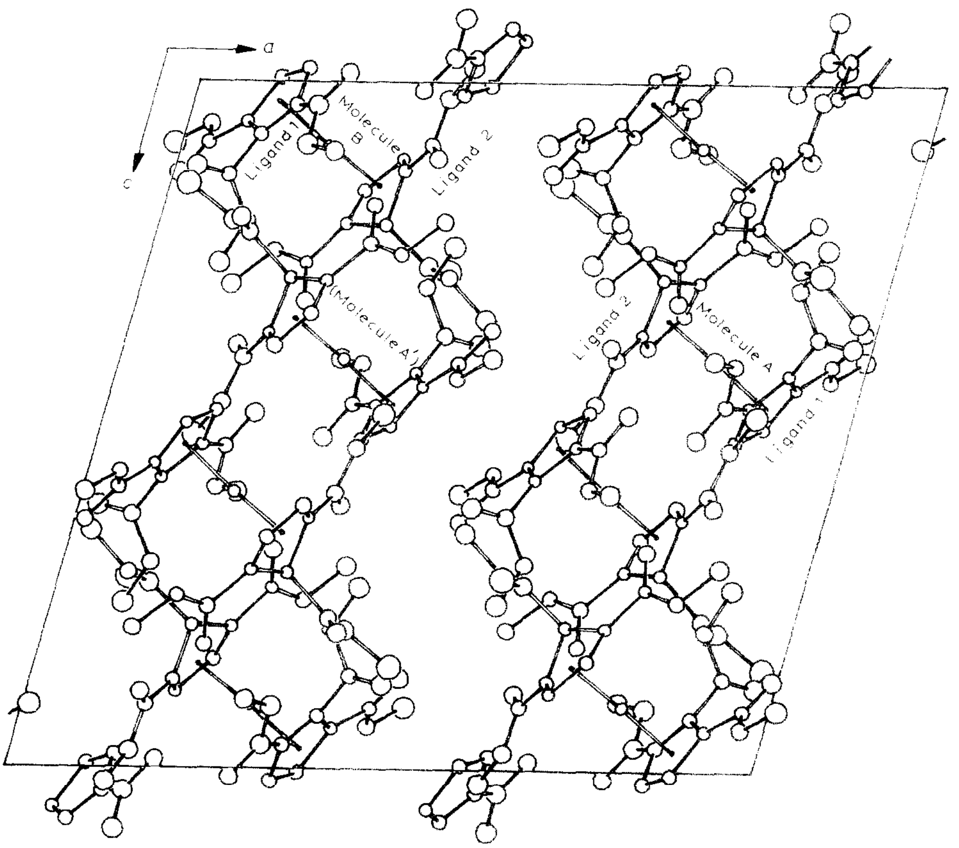


Fig. 2. The unit cell of 5, projected down *b*. Note the pseudo-symmetry, evident in the relationship of molecule B to molecule A, and also the pseudo-orthogonality of the cell diagonals.

mode. 8342 independent reflections were measured, 3053 with $I > 3\sigma(I)$ being considered 'observed' and used in block diagonal least-squares refinement without absorption correction after solution by vector methods. Anisotropic thermal parameters were refined for the metal atoms only; for the other non-hydrogen atoms, the

Table 1

Non-hydrogen atom parameters

Atom	Molecule A				Molecule B			
	x	y	z	$10^3\ U$ Å^2	x	y	z	$10^3\ U$ Å^2
Rh	0.7982(1)	0.0019(2)	0.4034(1)	[a]	0.1981(1)	−0.0683(2)	0.0933(1)	[a]
Ligand 1 (cp)								
C(1)	0.858(1)	0.124(2)	0.470(1)	26(6)	0.139(1)	0.056(2)	0.035(1)	38(7)
C(11)	0.827(1)	0.221(2)	0.469(1)	40(8)	0.167(2)	0.144(2)	0.035(2)	67(10)
O(11)	0.800(1)	0.251(2)	0.517(1)	62(6)	0.197(1)	0.179(1)	−0.013(1)	55(6)
O(12)	0.825(1)	0.273(1)	0.409(1)	47(6)	0.168(1)	0.200(2)	0.091(1)	73(7)
C(12)	0.795(2)	0.373(2)	0.403(2)	70(10)	0.206(2)	0.296(2)	0.101(2)	64(9)
C(2)	0.900(1)	0.087(2)	0.423(1)	26(6)	0.102(1)	0.024(2)	0.077(1)	36(7)
C(21)	0.925(2)	0.143(2)	0.370(2)	51(9)	0.074(1)	0.071(2)	0.135(1)	40(7)
O(21)	0.974(1)	0.198(2)	0.382(1)	71(7)	0.028(1)	0.133(2)	0.123(1)	71(7)
O(22)	0.882(1)	0.133(1)	0.299(1)	52(6)	0.116(1)	0.054(1)	0.204(1)	52(6)
C(22)	0.904(2)	0.189(3)	0.240(2)	79(12)	0.095(2)	0.105(2)	0.264(2)	73(11)
C(3)	0.914(1)	−0.019(2)	0.443(1)	22(6)	0.079(1)	−0.083(2)	0.059(1)	29(7)
C(31)	0.953(1)	−0.077(2)	0.409(1)	38(7)	0.035(1)	−0.152(2)	0.089(1)	35(7)
O(31)	0.986(1)	−0.049(1)	0.361(1)	46(5)	0.008(1)	−0.120(2)	0.133(1)	66(7)
O(32)	0.957(1)	−0.173(1)	0.431(1)	41(5)	0.032(1)	−0.239(1)	0.062(1)	50(6)
C(32)	0.997(2)	−0.241(2)	0.402(2)	65(10)	−0.010(2)	−0.305(3)	0.090(2)	84(12)
C(4)	0.887(1)	−0.041(2)	0.502(1)	24(6)	0.108(1)	−0.104(1)	−0.006(2)	35(7)
C(5)	0.852(1)	0.043(2)	0.520(1)	31(7)	0.141(1)	−0.017(2)	−0.019(1)	36(7)
C(0)	0.881(–)	0.038(–)	0.471(–)	–	0.114(–)	−0.024(–)	0.029(–)	–
Ligand 2 (diene)								
C(1)	0.746(1)	−0.032(2)	0.289(1)	28(7)	0.250(1)	−0.100(2)	0.207(1)	27(7)
C(11)	0.790(1)	−0.041(2)	0.235(1)	44(8)	0.211(1)	−0.112(2)	0.264(1)	28(7)
O(11)	0.780(1)	0.011(1)	0.180(1)	42(5)	0.224(1)	−0.060(2)	0.320(1)	55(6)
O(12)	0.835(1)	−0.115(1)	0.251(1)	37(5)	0.164(1)	−0.185(1)	0.245(1)	44(5)
C(12)	0.882(2)	−0.127(2)	0.204(2)	52(9)	0.120(2)	−0.200(2)	0.295(2)	64(10)
C(2)	0.697(1)	0.052(2)	0.286(1)	29(6)	0.303(1)	−0.014(2)	0.209(1)	29(6)
C(21)	0.624(2)	0.038(2)	0.220(1)	50(9)	0.376(2)	−0.027(2)	0.269(2)	60(10)
O(21)	0.607(1)	−0.040(2)	0.198(1)	66(7)	0.406(1)	−0.105(2)	0.278(1)	109(9)
O(22)	0.598(1)	0.127(2)	0.202(1)	71(7)	0.408(1)	0.051(1)	0.285(1)	49(5)
C(22)	0.525(2)	0.123(3)	0.150(2)	99(14)	0.473(2)	0.044(2)	0.346(2)	70(11)
C(3)	0.688(1)	0.034(2)	0.366(1)	23(6)	0.308(1)	−0.021(1)	0.129(1)	33(7)
C(31)	0.663(2)	0.115(2)	0.402(2)	49(8)	0.336(1)	0.047(2)	0.092(1)	41(8)
O(31)	0.651(1)	0.202(1)	0.384(1)	57(6)	0.348(1)	0.134(2)	0.111(1)	56(6)
O(32)	0.651(1)	0.083(1)	0.467(1)	53(6)	0.340(1)	0.014(2)	0.025(1)	60(6)
C(32)	0.627(2)	0.158(3)	0.515(2)	88(13)	0.361(2)	0.089(3)	−0.021(2)	72(11)
C(4)	0.700(1)	−0.073(2)	0.383(1)	45(8)	0.301(1)	−0.131(2)	0.112(1)	25(6)
C(5)	0.737(1)	−0.112(2)	0.338(1)	39(8)	0.262(1)	−0.177(2)	0.159(1)	35(7)
C(0)	0.718(–)	−0.046(–)	0.345(–)	–	0.280(–)	−0.108(–)	0.152(–)	–

[a] $10^3_{11,22,33,12,13,23}$ (Å^2): 29(1), 33(1), 13(1), −2(2), 4(1), −1(1) (Rh(A))
30(1), 32(1), 13(1), −3(1), 5(1), 0(1) (Rh(B))

Table 2

Rhodium environments

Distance (Å)	Molecule A		Molecule B	
	Ligand 1	Ligand 2	Ligand 1	Ligand 2
Rh–C(1)	2.23(2)	2.17(2)	2.18(3)	2.16(2)
Rh–C(2)	2.24(2)	2.65(2)	2.21(3)	2.68(2)
Rh–C(3)	2.21(2)	2.13(2)	2.26(2)	2.17(2)
Rh–C(4)	2.26(2)	2.12(3)	2.26(2)	2.13(2)
Rh–C(5)	2.24(2)	2.14(3)	2.22(2)	2.11(2)
Rh–C(0) [a]	1.85(–)	1.79(–)	1.87(–)	1.78(–)

[a] For ligand 2, C(0) is at the centre of gravity of C(1,3,4,5). C(10)–Rh–C(20) are 173.2, 177.6° for molecule A, B respectively. Deviations of C(2) from the C(1,3,4,5) plane for ligands 2 respectively are 0.47, 0.58 Å. Dihedral angles of $CO_2.C$ substituted plane to C(1,2,3,4.5) (ligand 1) or C(1,3,4,5) (ligand 2) planes at C(*n*) for molecules A, B respectively are

n	Ligand 1	Ligand 2
1	13.2; 7.8	26.3; 30.6
2	81.8; 78.1	83.2; 86.0
3	7.3; 9.4	12.0; 15.4

Dihedral angles between ligand 1 and 2 planes are 3.0, 8.9° for molecules A, B.

isotropic form was used. $(x, y, z, U_{\mathrm{iso}})_{\mathrm{H}}$ were included, constrained at estimated values. R, R' on $|F|$ at convergence were 0.090, 0.092, statistical reflection weights were derived from $\sigma^2(I) = \sigma^2(I)_{\mathrm{diff}} + 0.0001\sigma^4(I)_{\mathrm{diff}}$. Neutral complex scattering factors were used [8]; computation used the XTAL program system [9] implemented by S.R. Hall on a Perkin–Elmer 3240 computer.

Crystal data. $\mathrm{Rh\{C_5H_3(CO_2Me)_3\}\{C_5H_2(CO_2Me)_3\}} = \mathrm{C_{22}H_{23}O_{12}Rh}$, M = 582.3, Monoclinic, space group $P2_1/a$ (C_{2h}^5, No. 14), a 19.543(7), b 13.52(1), c 18.789(7) Å, β 105.10(3)°, U 4794(5) Å^3. D_c ($Z = 8$) 1.61 g cm^{-3}. $F(000) = 2368$. μ_{Mo} 7.3 cm^{-1}. Specimen 0.10 × 0.10 × 0.48 mm (needle).

Abnormal features. The conformations of the two independent molecules are identical; atom labelling is similar for similar atoms. In the CO.OMe groups attached to ring atoms *n*, atoms are labelled CO(*n*1).OC(*n*2). Pseudo-symmetry is evident in the structure (Fig. 2); reflections (*h*0*l*) are present only for *h*, *l* both even. Assignment of space group was resolved only after attempted solution of the structure in all of $P2_1/a$, *n* and *c*.

Non-hydrogen atom parameters for the two independent molecules and rhodium environments are given in Tables 1 and 2, respectively. A table of thermal parameters and a list of observed and calculated structure factors are available from the authors.

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