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Electron density manipulation in rhodium(I) phosphine complexes: structure of acetylacetonatocarbonylferrocenyldiphenylphosphinerhodium(I)

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Abstract—The title complex [Rh(acac)(CO)(PPh_2Fc)] (acac = acetylacetonato, PPh_2Fc = ferrocenyldiphenylphosphine), was obtained in acetone by reaction of the tertiary phosphine with [Rh(acac)$(CO)_2$] and was characterised by IR spectroscopy, ^1H and ^{31}P NMR and single crystal X-ray crystallography. This is a rare example illustrating the incorporation of the ferrocenyl moiety in a monodentate tertiary phosphine complex of rhodium(I). It crystallises in the triclinic space group $P_{\bar{1}}$ with two independent molecules in the asymmetric unit and refined to R = 2.40% from 5215 reflections. The independent molecules, showing the usual square planar geometry, differ mainly in the rotation mode of the asymmetric phosphine around the Rh-P bond and a slight deviation in the Rh-CO bond from linearity, which was correlated with IR data. © 1998 Elsevier Science Ltd. All rights reserved

Keywords: structure; characterisation; rhodium; ferrocene; phosphine

The structure of the title compound was determined in the wider context of research done by our group on [Rh(β-dik)(CO)(PR_3)] complexes [1] (β-dik = β-diketone; PR_3 = tertiary phosphine ligands). These complexes are especially well known for the oxidative addition studies performed on them [2]. Investigations on symmetrical and unsymmetrical bidentate ligands, the effect of different bidentate ligand donor atoms bonded to the metal centre as well as different phosphine and phosphite ligands and solvent effects were some of the studies performed by our group. Furthermore it was shown that Rh-P coupling constants obtained from ^{31}P NMR studies can give some more valuable information about the character of the Rh-P bond and correlations between bond lengths, bite angle of the bidentate ligand and reactivity were made [3].

The ferrocenyl group is a convenient moiety to introduce as part of the ligand system (very good σ-donor) of these Rh(I) complexes since it provides a convenient tool to study, especially by electrochemical methods, the electronic characteristics of Rh(I) or Rh(III) compounds. Current research has revealed a direct correlation between the Lewis basicity of the central rhodium atom and the oxidation potential of the ferrocenyl unit attached to the β-diketone in complexes of the type [Rh(β-dik)$(P(OPh)_3)_2$] and [Rh(β-dik)(COD)] where β-dik = ferrocenylacetonato, ferrocenyltrifluoroacetonato and diferrocenylacetonato and COD = cis-1,5-cyclooctadiene [4].

In this paper we report the crystal and molecular structure of acetylacetonatocarbonylferrocenyldiphenylphosphinerhodium(I), which has been determined to gain additional information on the bonding mode and steric demand of this very crystalline unsymmetrical phosphine ligand, PPh_2Fc. This forms part of a programme to investigate the effect thereof on selected aspects of organometallic platinum group metal complexes. A common platinum(II) compound containing this phosphine ligand, *trans*-dichlorobis(ferrocenyldiphenylphosphine)platinum(II), was also prepared as part of this platinum group metal project and the structure was already published [5]. For the tuning of the reactivity of organometallic complexes a thorough understanding of the characteristics

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of the different ligands employed in the system is of crucial importance. The structural information obtained from this study is compared with that of related complexes and correlated with its NMR and IR parameters.

EXPERIMENTAL

Physical measurements

IR spectra were recorded on a Hitachi 270–50 spectrometer in KBr pellets in the region 4000–250 cm^{-1} or as a 0.0052 M solution in $CHCl_3$ in the region 2200–1650 cm^{-1}. NMR [^{1}H (300 MHz), ^{31}P{^{1}H} (121.497 MHz), 25°C] spectra were recorded on a Bruker 300 MHz spectrometer in $CDCl_3$ and chemical shifts are reported relative to the residual $CHCl_3$ peak (7.24 ppm) for the ^{1}H spectra and relative to 85% H_3PO_4 (0 ppm) for the ^{31}P spectra (positive shifts downfield).

Preparation

PPh$_2$Fc. Prepared according to literature procedures [6] and purified by column chromatography (Hexane:Benzene, 9:1). NMR, δ (ppm): ^{1}H: 4.06 (s, 5H); 4.09 (q, 2H); 4.36 (t, 2H); 7.3–7.4 (m, 10H); ^{31}P: −16.18 (s).

[*Rh*(*acac*)(*CO*)$_2$]. To a stirred solution of [Rh(μ-Cl)(CO)$_2$]$_2$ (51 mg; 0.131 mmol) in freshly distilled DMF (2 ml) was added acetylacetone (28 mg; 0.275 mmol) and stirring was continued for a further 2 min before the reaction mixture was quenched with ca 80 ml of distilled water. The strawberry red precipitate obtained was isolated by centrifugation and recrystallised from hot acetone as fine dark green needles (48 mg; yield = 80%). NMR, δ (ppm): ^{1}H: 2.09 (s, 6H); 5.63 (s, 1H).

[*Rh*(*acac*)(*CO*)(*PPh*$_2$Fc)]. Prepared by the slow treatment of a stirred acetone solution (4 ml) of the freshly prepared [Rh(acac)(CO)$_2$] (48 mg; 0.19 mmol) with solid PPh$_2$Fc (77 mg; 0.208 mmol) resulting in the product as a fine orange precipitate in reasonable yields (ca 70%). Bright orange medium quality crystals, suitable for X-ray analysis, were obtained by the slow evaporation of a concentrated acetone solution. NMR , δ (ppm): ^{1}H: 1.67 (s, 3H); 2.14 (s, 3H); 4.26 (s, 5H); 4.43 (m, 2H); 4.47 (m, 2H); 5.46 (s, 1H); 7.3–7.7 (m, 10H); ^{31}P: 42.3 (d), $^1J_{\text{Rh-P}} = 177.3$ Hz.

Crystal structure

An orange cube of the title compound with dimensions of 0.20 × 0.25 × 0.25 mm was mounted on an Enraf Nonius CAD 4 four circle diffractometer. Graphite monochromated Mo-K_α radiation ($\lambda = 0.71073$ Å) and a θ-2θ scan type were used to measure the intensity data in the range $1.71 \leq \theta \leq 25.05$ at a temperature of 293(2) K. The unit cell was determined and refined from 25 reflections in the θ range 3.24–23.87°. Crystal data for [Rh(acac)(CO)(PPh$_2$Fc)]: $C_{56}H_{52}O_6P_2Fe_2Rh_2$, triclinic, space group $P\bar{1}$, $a = 12.460(2)$, $b = 14.052(3)$, $c = 16.325(3)$ Å, $\alpha = 105.96(3)$, $\beta = 110.52(3)$, $\gamma = 95.15(3)$°, $V = 2518.2(8)$ Å, $Z = 2$, $D_c = 1.583$ g cm^{-3}, $D_m = 1.547$ g cm^{-3} (aqueous NaI), μ(Mo-K_α) = 1.323 mm^{-1}, $F(000) = 1216$, *hkl* ranges $-13 \leq h \leq 14$, $-15 \leq k \leq 16$, $-18 \leq l \leq 0$. The 5224 independent reflections collected of 5418 measured reflections were corrected for Lorentz and polarisation effects and empirical absorption corrections were done on the faced crystal. A correction was also applied for decay (0.3%) which was determined by monitoring of three standards every 100 reflections. Data reduction was done by Profit [7]. The structure was solved by the heavy atom method and refined by full-matrix least-squares calculations respectively using the SHELXS86 and SHELXL93 series of programmes [8]. Refinement converged to a final $R = 0.0240$ (for 5215 with $F_o > 4\sigma(F_o)$, $wR2 = 6.20$, for all unique 5223 data, 1 suppressed, 626 parameters), with allowance for thermal anisotropy for all non-hydrogen atoms. The positions of the methine protons were determined from a difference Fourier map and refined isotropically. Other hydrogen atoms were calculated in the idealised positions riding on the adjacent carbon atom and refined with an overall isotropic thermal parameter. A weighting scheme $w = 1/[\sigma^2(F_o^2) + (0.0294^*P)^2 + 3.39^*P]$, where $P = (F_o^2 + 2^*F_c^2)/3$, was used in the latter stages of the refinement. The maximum and minimum peaks on the final difference Fourier map, and goodness-of-fit corresponded to 0.416 and −0.394 eÅ^{-3}, and 1.066 respectively. The graphics were done with ORTEP II [9]. A summary of selected bond lengths, angles and torsion angles for molecules 1 and 2 is given in Table 1.

RESULTS AND DISCUSSION

Description of the structure of [*Rh*(*acac*)(*CO*)(*PPh*$_2$*Fc*)]

Both molecules crystallise with slightly distorted square planar geometry around the central Rh atoms, see Fig. 1. The closest contact between different Rh centres of the two Rh entities is 6.7 Å, suggesting no significant intermolecular Rh-Rh interactions. The difference in the trans influence of the phosphine and carbonyl groups is obvious from the difference in the Rh-O bond distances of 0.045 Å and 0.030 Å for molecules 1 and 2 respectively, the longer bond corresponding to the one trans to the PPh$_2$Fc ligand.

The phosphorus atoms are tetrahedrally surrounded by the two phenyl rings, the ferrocenyl ligand and the Rh atom with the C-P-Rh bonds all larger than the ideal 109.5°, ca 110–117°, while all C-P-C angles are smaller than average, at ca 102–106°. The deviation from normal tetrahedral angles towards smaller values

Table 1. Bond lengths (Å), angles (°) and tortion angles (°) for molecule 1 and 2 with e.s.d.'s in parentheses

Molecule 1		Molecule 2	
Rh(1)-C(16)	1.799(4)	Rh(2)-C(26)	1.800(4)
Rh(1)-O(11)	2.027(3)	Rh(2)-O(21)	2.038(3)
Rh(1)-O(12)	2.072(3)	Rh(2)-O(22)	2.068(3)
Rh(1)-P(1)	2.2331(11)	Rh(2)-P(2)	2.2375(13)
P(1)-C(111)	1.830(4)	P(2)-C(211)	1.824(4)
P(1)-C(121)	1.824(4)	P(2)-C(221)	1.828(4)
P(1)-C(131)	1.808(4)	P(2)-C(231)	1.805(4)
O(11)-C(12)	1.270(5)	O(21)-C(22)	1.265(5)
O(12)-C(14)	1.279(5)	O(22)-C(24)	1.261(5)
O(13)-C(16)	1.144(5)	O(23)-C(26)	1.146(5)
C(11)-C(12)	1.495(6)	C(21)-C(22)	1.504(6)
C(12)-C(13)	1.383(7)	C(22)-C(23)	1.378(7)
C(13)-C(14)	1.383(7)	C(23)-C(24)	1.380(7)
C(14)-C(15)	1.497(6)	C(24)-C(25)	1.510(7)
O(11)-Rh(1)-O(12)	88.09(12)	O(21)-Rh(2)-O(22)	88.04(12)
O(11)-Rh(1)-P(1)	88.73(9)	O(21)-Rh(2)-P(2)	90.58(9)
O(12)-Rh(1)-P(1)	174.57(9)	O(22)-Rh(2)-P(2)	178.54(9)
C(16)-Rh(1)-O(11)	176.0(2)	C(26)-Rh(2)-O(21)	176.3(2)
C(16)-Rh(1)-O(12)	93.6(2)	C(26)-Rh(2)-O(22)	91.7(2)
C(16)-Rh(1)-P(1)	89.86(13)	C(26)-Rh(2)-P(2)	89.68(14)
O(13)-C(16)-Rh	177.3(4)	O(23)-C(26)-Rh(2)	175.2(4)
C(2&4)-O(1&2)-Rh(1)$_{avg}$	127.3(3)	C(2&4)-O(1&2)-Rh(2)$_{avg}$	127.2(3)
(C-P-Rh)$_{avg}$	114.32(13)	(C-P-Rh)$_{avg}$	114.5(2)
(C-P-C)$_{avg}$	104.1(2)	(C-P-C)$_{avg}$	103.9(2)
O(11)-C(12)-C(13)	125.3(4)	O(21)-C(22)-C(23)	124.8(4)
O(11)-C(12)-C(11)	114.5(4)	O(21)-C(22)-C(21)	115.0(4)
O(12)-C(14)-C(13)	125.8(4)	O(22)-C(24)-C(23)	125.2(5)
O(12)-C(14)-C(15)	114.9(4)	O(22)-C(24)-C(25)	115.1(4)
C(12)-C(13)-C(14)	126.2(4)	C(22)-C(23)-C(24)	127.3(5)
C(13)-C(12)-C(11)	120.1(4)	C(23)-C(22)-C(21)	120.2(4)
C(13)-C(14)-C(15)	119.3(4)	C(23)-C(24)-C(25)	119.6(5)
C132-C131-C141-C142	-4.6(4)	C232-C231-C241-C242	-5.3(4)
C133-C132-C142-C143	-5.2(4)	C233-C232-C242-C243	-5.0(5)
C134-C133-C143-C144	-4.4(4)	C234-C233-C243-C244	-5.9(4)
C135-C134-C144-C145	-5.9(5)	C235-C234-C244-C245	-5.9(5)
C131-C135-C145-C141	-7.1(4)	C231-C235-C245-C241	-6.2(4)
O11-Rh1-P1-C111	-108.5(2)	O21-Rh2-P2-C211	-111.2(2)
O11-Rh1-P1-C121	10.7(2)	O21-Rh2-P2-C221	7.5(2)
O11-Rh1-P1-C131	126.2(2)	O21-Rh2-P2-C231	123.3(2)
C132-C131-P1-Rh1	1.2(4)	C232-C231-P2-Rh2	-10.9(4)
C141-C131-P1-C111	-37.3(2)	C241-C231-P2-C211	-46.5(2)

for the C-P-C angles in phosphine ligands can be explained on the basis of the valence-shell electron-pair repulsion theory where the phosphorous lone pair will repel the bonding pair electrons to a greater extent than they will repel each other. Greater deviation from normal tetrahedral angles towards smaller values are then normally an indication of more lone pair character on the phosphorous atom. Upon coordination the lone pair becomes a bonding pair, resulting in weakened repulsion and the angles become closer to the expected value. This observation also holds true in the current study where coordination gives rise to an average increase of 3.0(2)° in the C-P-C angles compared with those in the uncoordinated phosphine [10].

The P-C bond distances to the phenyl rings are within normal limits for this type of bond, while the P-C bond for the ferrocenyl moiety is appreciably shorter by 0.032(8) Å. The same tendency was also previously observed in structural studies containing the PPh$_2$Fc ligand [5]. This observation can be attributed to the better electron donating capability of the ferrocenyl moiety compared to phenyl, giving rise to a shorter P-C bond.

The Fe-C bond lengths are all within normal ranges for ferrocene compounds, averaging 2.037(5) Å for both the substituted and unsubstituted rings in molecules 1 and 2. Selected torsion angles are given in Table 2 and show the cyclopentadienyl rings for

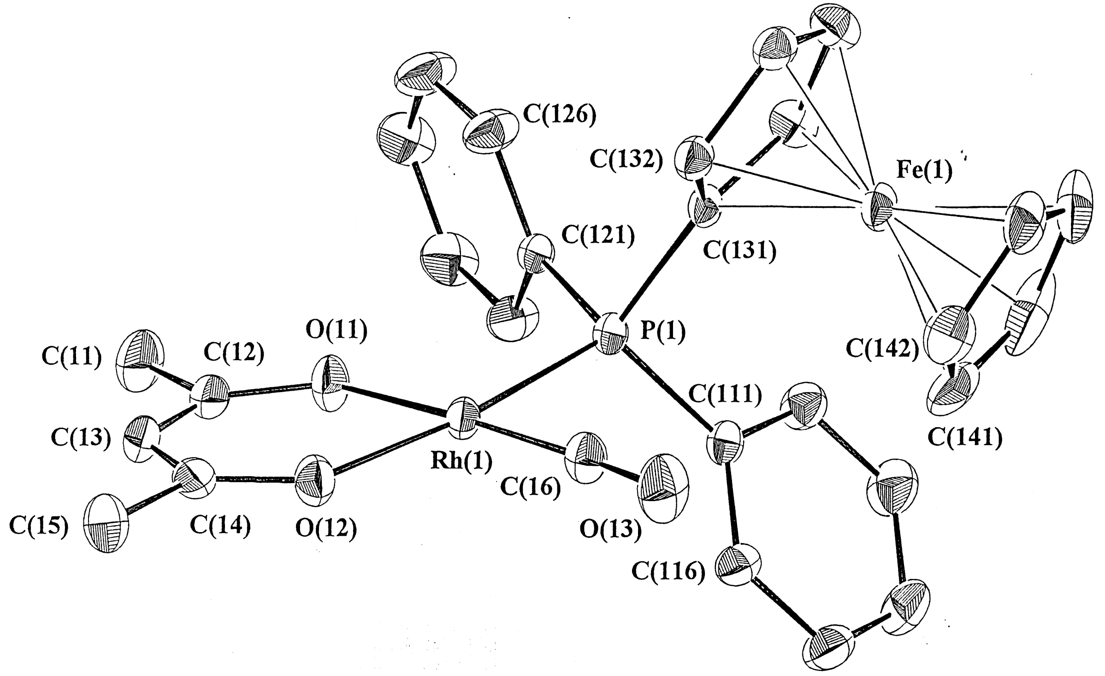

Fig. 1. An ORTEP II drawing of molecule 1 of the coordination compound showing the numbering scheme. The first digit refers to molecule 1 or 2 and the second to the number of the atom in the molecule. Displacement ellipsoids correspond to 30% probability level. Hydrogen atoms are omitted for clarity.

Table 2. Comparisons of Rh(β-Dik)(CO)(PR$_3$) complexes with e.s.d.'s in parentheses

	Rh(acac)(CO)(PPh$_2$Fc)[a]	Rh(acac)(CO)(PPh$_3$)	Rh(3-Bz-acac)(CO)(PPh$_3$)
Rh-C(6)	1.800(4)	1.801(8)	1.801(5)
Rh-O(1)	2.033(3)	2.029(5)	2.048(3)
Rh-O(2)	2.070(3)	2.087(4)	2.070(3)
Rh-P(1)	2.2353(12)	2.244(2)	2.232(1)
O(1)-C(2)	1.268(5)	1.274(7)	1.266(5)
O(2)-C(4)	1.270(5)	1.275(5)	1.264(5)
O(3)-C(6)	1.145(5)	1.153(11)	1.147(5)
O(1)-Rh-O(2)	88.07(12)	87.9(2)	88.6(1)
O(1)-Rh-P	89.66(9)	92.0(1)	85.9(1)
C(6)-Rh-O(2)	92.7(2)	92.4(2)	92.9(2)
C(6)-Rh-P	89.66(14)	87.8(2)	92.7(1)
O(3)-C(6)-Rh	177.3(4), 175.2(4)	176.8(9)	178.1(4)
ν(CO) (cm^{-1})			
KBr	1974, 1958	1983	1959
CHCl$_3$	1980	1984	1982
^{31}P{^{1}H}			
δ(P)/ppm	42.3	48.99	49.44
$^1J_{\text{Rh-P}}$/Hz	177.3	173.0	175.0

[a] Average for molecule 1 and 2.

both molecules to be in almost an eclipsed conformation.

An important difference between the independent molecules lies in the rotation of the ferrocenyl moiety relative to the coordination plane where a difference of almost 10° is observed. A difference in rotation around the Rh-P bond for the different molecules of almost 3° enhances this effect (Fig. 3).

The Rh-P bond distance in the title compound compared with the analogous [Rh(acac)(CO)(PPh$_3$)] [11]

complex is slightly shorter, indicating stronger Lewis basicity, as one would expect the larger steric demand of the PPh_2Fc ligand to give rise to an increase in the Rh-P bond. It is however about the same length as the Rh-P bond found in the [Rh(3-Bz-acac)(CO)(PPh_3)] [12] (3-Bz-acac = 3-benzyl-acetylacetonato) complex.

IR and NMR Spectra

The results of the IR and NMR investigations are summarised in Table 2. Certainly the most important aspect to note from the IR spectra for the title compound is the presence of two carbonyl stretching frequencies at 1974 and 1958 cm^{-1} (KBr discs) respectively for the two independent molecules. The presence of these two peaks in the solid state was one of the interesting discoveries made upon isolation of the crystalline title compound since [Rh(β-dik)(CO)(PR_3)] complexes are usually characterised by a single carbonyl stretching frequency. Thus, additional characterisation beyond that of normal IR spectroscopy was deemed necessary. Solution IR (single $\nu(CO) = 1980\,cm^{-1}$ in $CHCl_3$) and NMR results showed only the expected compound and suggested two 'packing isomers' to be present in the crystalline solid state. (Fig. 2).

Upon solution of the molecular structure by X-ray crystallography this was indeed revealed and a significant difference of 2.1(4)° was observed in the Rh-C-O angles for the two independent molecules giving rise to the difference of 16 cm^{-1} in the ν(CO) values. No significant difference in either the Rh-C nor the C-O bond lengths in the Rh-CO moieties, indicating a difference in Rh-C or C-O bond strength, could be observed for either of the molecules. This is however to be expected given the usual accuracy by which the individual atoms in a carbonyl group can be determined by X-ray crystallography at ambient temperature. The observed difference in ν(CO) therefore originates from electronic factors induced by packing effects. These packing forces induces a bent mode in the Rh(2)-C(26)-(O23) bond (175.2(4)° molecule 2) compared to the Rh(1)-C(16)-O(13) bond (177.3(4)° molecule 1), also see Fig. 3. This gives rise to a decrease in *sp* character in the carbonyl moiety in molecule 2, which in turn is reflected in the corresponding decrease in the ν(CO) value of 1958 cm^{-1} compared to the ν(CO) value of 1974 cm^{-1} for molecules 2 and 1 respectively. Motivation for this assignment comes from a previous solid state IR and X-ray crystallographic investigation [13] of the Rh analogue of Vaska's complex, [Rh(PPh_3)$_2$(CO)(Cl)], wherein the Rh-CO moiety (as determined from two different polymorphs) exhibited angles of 177.2(4)° and 164(4)° respectively, with corresponding ν(CO) values of 1983 cm^{-1} and 1965 cm^{-1}. However, in the case of the [Rh(3-Bz-acac)((CO)PPh_3)] complex there is not as good a correlation as in the above mentioned examples (Rh-CO angle of 177.8(4) and ν(CO) 1959 cm^{-1}). A more detailed investigation to these effects is currently being undertaken on a broad spectrum of metal carbonyl complexes.

Upon comparison of the respective ν(CO) values obtained in solution (although they only differ slightly) for the title compound, [Rh(3-Bz-acac)(CO)(PPh_3)] and [Rh(acac)(CO)(PPh_3)] of 1980 cm^{-1}, 1982 cm^{-1} and 1984 cm^{-1} respectively (listed in Table 2) suggests that the metal atom with the highest electron density to be that of the title compound, secondly the 3-Bz-acac/PPh_3 complex and thirdly the acac/PPh_3

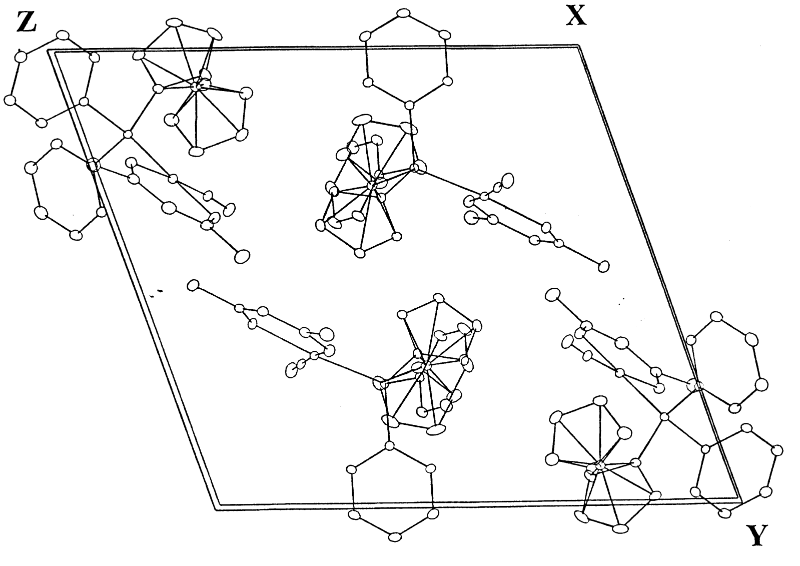

Fig. 2. Packing diagram of the unit cell showing the relative orientation of the two independent molecules in the unit cell. Hydrogen atoms are omitted for clarity. Displacement ellipsoids are at arbitrary size.

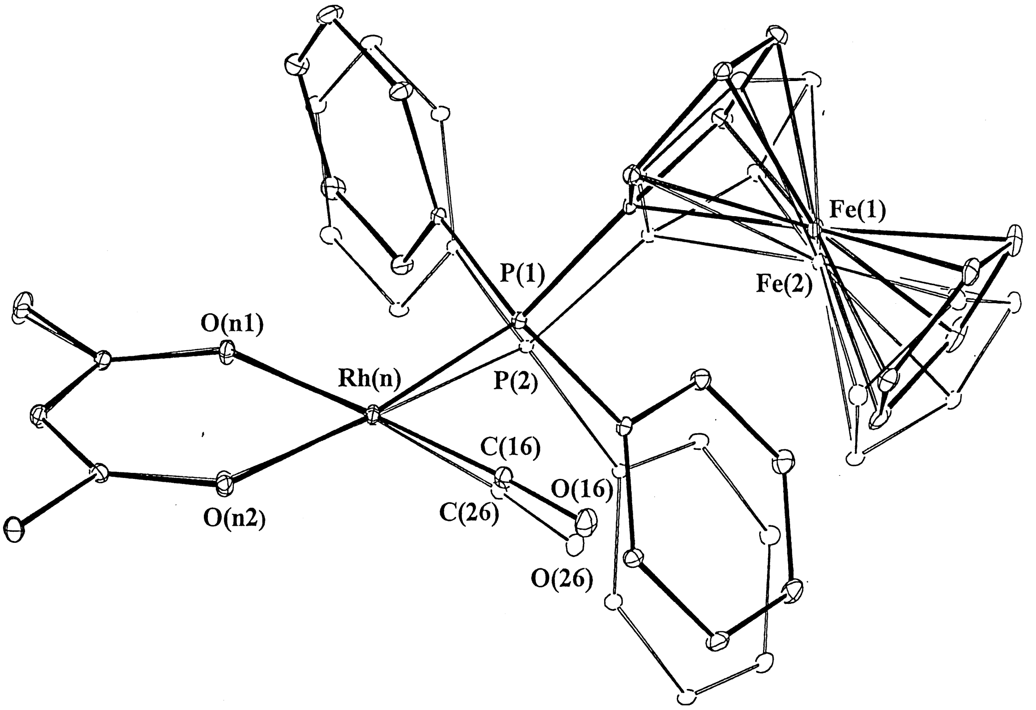

Fig. 3. Molecules 1 and 2 superimposed on each other showing the subtule differences due to packing effects. Hydrogen atoms are omitted for clarity. Displacement ellipsoids are at arbitrary size. The solid bonds correspond to molecule 1 and the open bonds to molecule 2.

complex. This conclusion is also confirmed by the NMR results obtained. The ^{31}P chemical shifts for the acac/PPh$_3$ and 3-Bz-acac/PPh$_3$ complexes are within 0.4 ppm, suggesting almost equal electronic environments for the respective P atoms, the 3-Bz-acac/PPh$_3$ complex being the more shielded of the two (upfield shift). One would expect an increase in electron density on the Rh centre established by the better electron donating capability of the 3-Bz-acac ligand compared to acac. The ^{31}P chemical shift for the acac/PPh$_2$Fc complex is 7 ppm further upfield, indicating a substantial increase in the electronic shielding on the P atom of the PPh$_2$Fc ligand in comparison with the PPh$_3$ ligand in the other two mentioned complexes. First order coupling between the Rh and P atoms arising from orbital interactions between the respective nuclei suggests the most effective interaction to be that of the PPh$_2$Fc ligand (177.3 Hz), the 3-Bz-acac/PPh$_3$ complex second (175.0 Hz) and the acac/PPh$_3$ complex the least effective (173.0 Hz). The magnitude of coupling constants can to a great extent be correlated successfully with the bond lengths obtained in similar systems [3]. This is also reflected in the present case where the Rh-P bond distances for the acac/PPh$_2$Fc and 3-Bz-acac/PPh$_3$ complexes are the same in length and that of the acac/PPh$_3$ complex is a bit longer, resulting in the smaller coupling constant thereof.

This study enabled a well defined characterisation of the title compound, showing several interesting aspects. The effect of the PPh$_2$Fc ligand in corresponding reactivity studies of Rh(I), Pt(II) and Pd(II) complexes will be investigated in future.

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