

Refinement

Refinement on F	$(\Delta/\sigma)_{\max} = 0.007$
$R = 0.0208$	$\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
$wR = 0.0165$	$\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$
$S = 1.577$	Extinction correction: none
807 reflections	Atomic scattering factors
94 parameters	from <i>International Tables</i>
All H-atom parameters	for <i>Crystallography</i> (1992),
refined	Vol C, Tables 4.2.6.8 and
$w = 4F_o^2/[\sigma^2(F_o^2)$	6.1.1.4)
$+ (0.003F_o^2)^2]$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$)

	x	y	z	U_{eq}
Ru(1)	0	0	0	0.0320 (1)
Cl(1)	0.06866 (8)	0.1215 (1)	0.15448 (7)	0.0452 (3)
N(1)	0.0684 (2)	-0.2059 (3)	0.0436 (2)	0.0366 (9)
N(2)	-0.1402 (2)	-0.0682 (3)	0.0768 (2)	0.0363 (9)
C(1)	0.0955 (3)	-0.3246 (4)	0.0714 (3)	0.039 (1)
C(2)	0.1287 (4)	-0.4784 (6)	0.1094 (4)	0.057 (2)
C(3)	-0.2097 (3)	-0.1180 (4)	0.1256 (3)	0.038 (1)
C(4)	-0.2978 (4)	-0.1835 (6)	0.1922 (4)	0.053 (1)

Table 2. Selected geometric parameters ($\text{\AA}$, $^\circ$)

Ru(1)—Cl(1)	2.4101 (9)	N(2)—C(3)	1.123 (4)
Ru(1)—N(1)	2.021 (3)	C(1)—C(2)	1.461 (6)
Ru(1)—N(2)	2.020 (3)	C(3)—C(4)	1.466 (5)
N(1)—C(1)	1.125 (4)		
Cl(1)—Ru(1)—N(1)	90.39 (8)	Ru(1)—N(2)—C(3)	171.7 (3)
Cl(1)—Ru(1)—N(2)	90.92 (8)	N(1)—C(1)—C(2)	178.7 (5)
N(1)—Ru(1)—N(2)	93.7 (1)	N(2)—C(3)—C(4)	178.1 (4)
Ru(1)—N(1)—C(1)	172.7 (3)		

The θ -scan width was $(0.80 + 1.3 \tan \theta)^\circ$ at a speed of $4.0^\circ \text{ min}^{-1}$ (in ω). The weak reflections were rescanned a maximum of four times and the counts accumulated to ensure good counting statistics. Stationary background counts were made on each side of the reflection with a 2:1 ratio of peak-to-background counting time. H atoms were refined isotropically. The structure was solved using heavy-atom Patterson methods (Beurskens *et al.*, 1992) and expanded using Fourier techniques (Beurskens *et al.*, 1992).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *PATY* in *DIRDIF92* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

We wish to thank The Royal Society (UK) Australian Scheme for the award of a Postdoctoral Fellowship to MB, and Dr Graham Heath and Professor Martin Bennett for stimulating discussions and encouragement.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1049). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

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Acta Cryst. (1996). **C52**, 1106–1110

Two Monomeric Ruthenium Complexes Containing Bidentate Bis(diphenylphosphino) Ligands

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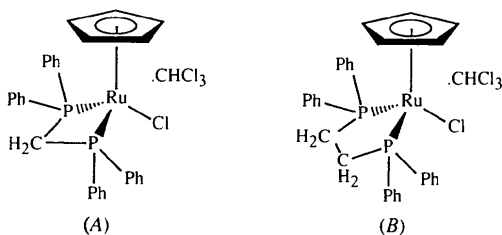
Abstract

The crystal structures of chloro(η^5 -cyclopentadienyl)-[methylenebis(diphenylphosphine-*P*)]ruthenium–chloroform (1/1), [RuCl(C₅H₅)(C₂₅H₂₂P₂)]·CHCl₃, (*A*), and chloro(η^5 -cyclopentadienyl)[1,2-ethanediylbis(diphenylphosphine-*P*)]ruthenium–chloroform (1/1), [RuCl(C₅H₅)(C₂₆H₂₄P₂)]·CHCl₃, (*B*), are reported. Both complexes contain a central ring structure in which a pair of P atoms, linked by a —CH₂— [in (*A*)] or a —C₂H₄— [in (*B*)] group, are bonded to a central Ru atom. The P—Ru—P bond angle undergoes expansion from 72.07 (2)° in (*A*) to 83.48 (2)° in (*B*). The bond distances around the Ru center are compared with values reported for

similar compounds. Both structures include a chloroform solvent molecule in addition to the ruthenium complex. The chloroform molecule in (A) was found to be disordered.

Comment

Ruthenium forms a variety of classical coordination and organometallic compounds. The two compounds reported here, (A) and (B), were initially isolated by evaporation of chloroform/pentane solutions containing by-products of the photolysis reactions of $(\eta^5\text{-C}_5\text{H}_5)_2\text{Ru}_2(\mu\text{-CO})_4$ with dpmp [bis(diphenylphosphino)methane] or dppe [1,2-bis(diphenylphosphino)ethane]. Both compounds were then independently prepared through the reaction of $(\eta^5\text{-C}_5\text{H}_5)\text{Ru}(\text{PPh}_3)_2\text{Cl}$ with dpmp or dppe in refluxing benzene following the procedure of Ashby, Bruce, Tomkins & Wallis (1979). Proton NMR spectra from the latter preparations were consistent with those of the photolysis products.



The structure of (B) with deuterated chloroform as a solvent has been reported previously (Suravajjala & Porter, 1993). A major difference between the earlier structure and the redetermination described here is that the phenyl rings were treated as rigid groups in the former, but were not constrained in this manner in this work. While the essential features of the two structures are the same, the e.s.d.'s for the structure described here are significantly lower than those of the previously reported structure.

The Ru—P distances in both compounds [2.2724 (5) and 2.2833 (6) Å in (A), 2.2688 (7) and 2.2863 (7) Å in (B)] are consistent with Ru—P distances in similar compounds. Ru—P distances range from 2.224 to 2.337 Å with an average value of 2.282 Å in a series of compounds containing the Cl—Ru—Cp fragment with a variety of phosphino ligands (Bruce, Wong, Skelton & White, 1981; de Klerk-Engels, Groen, Vrieze, Möckel, Lindner & Goubitz, 1992; Joslin, Mague & Roundhill, 1991; Mague & Johnson, 1990; Morandini, Consiglio, Straub, Ciani & Sironi, 1983). The central hydrocarbon bridge between the P atoms in both phosphino ligands appears to decrease the steric interaction between phenyl rings so that the Ru—P distances in (A) and (B) are closer to those found in the analogous bis(trimethylphosphino) complex ($\langle\text{Ru—P}\rangle$ 2.275 Å) than in

the bis(triphenylphosphino) complex ($\langle\text{Ru—P}\rangle$ 2.336 Å) (Bruce, Wong, Skelton & White, 1981). The bond lengths are in quite good agreement with those in the diphenylphosphinopropane complex ($\langle\text{Ru—P}\rangle$ 2.277 Å) (Morandini, Consiglio, Straub, Ciani & Sironi, 1983). The difference between the two Ru—P distances in compounds (A) and (B) most likely results from intermolecular interactions between the chloroform solvent and the phenyl groups on P1, as the solvent molecule is closely associated with these groups in both structures [as can be seen in Fig. 3 for (B)]. The corresponding Ru—P distances are quite similar in both compounds. The larger bite of the dppe ligand is accommodated by a larger P—Ru—P angle in (B) [83.48 (2)°] compared to that in (A) [72.07 (2)°].

Unequal Ru—C distances are found in both (A) and (B), similar to those in chloro(η^5 -cyclopentadienyl)-bis(trimethylphosphine)ruthenium [2.15 (2)–2.23 (2) Å; Bruce, Wong, Skelton & White, 1981], chloro(η^5 -cyclopentadienyl)bis(diphenylphosphinopropane)ruthenium [2.159 (9)–2.230 (10) Å; Morandini, Consiglio, Straub, Ciani & Sironi, 1983] and chloro(η^5 -cyclopentadienyl)[bis(dimethoxyphosphino)methylamine]ruthenium [2.183 (2)–2.254 (2) Å; Mague & Johnson, 1990]. In each case the shorter Ru—C distances are found *trans* to the chloro ligand, with longer Ru—C distances *trans* to the P atoms. This is consistent with the differing *trans* influences of Cl and P. The Ru—Cl distances [2.4302 (6) in (A), 2.4466 (7) Å in (B)] agree quite well with those in the same series of compounds (2.423–2.459, average 2.441 Å).

The average C—C distances in the phenyl rings are 1.379 (4) in (A) and 1.376 (4) Å in (B), with values ranging from 1.351 (5) to 1.393 (4) Å and from 1.339 (7) to 1.393 (5) Å in (A) and (B), respectively. Within the cyclopentadienyl ligands, average C—C distances are 1.389 (5) in (A) and 1.400 (4) Å in (B), with values ranging from 1.358 (5) to 1.419 (5) Å in (A) and from 1.390 (4) to 1.413 (4) Å in (B).

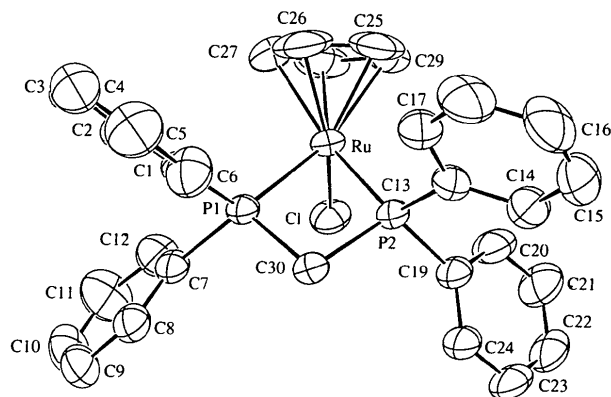


Fig. 1. ORTEP (Johnson, 1976) diagram of the ruthenium complex in (A). Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

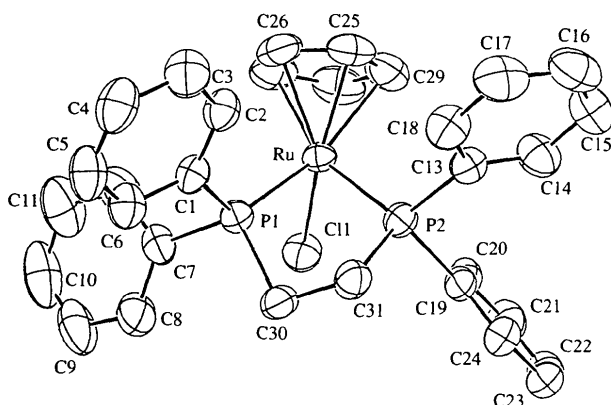


Fig. 2. ORTEP (Johnson, 1976) diagram of the ruthenium complex in (B). Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

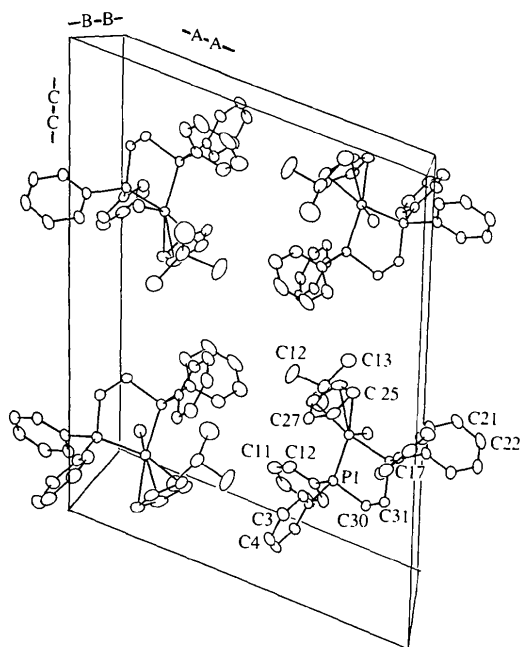


Fig. 3. The unit cell for (B) showing the orientation of chloroform solvent molecules.

Experimental

Compounds (A) and (B) were prepared by the evaporation of chloroform/pentane solutions resulting from the photolysis reactions of tetracarbonyl(η^5 -cyclopentadienyl)ruthenium dimer with bis(diphenylphosphino)methane and bis(diphenylphosphino)ethane, respectively. The density D_m of each was measured by flotation in salt solution followed by measurement of the mass of 10.0 ml of the solution.

Compound (A)

Crystal data

[RuCl(C₅H₅)(C₂₅H₂₂P₂)]·CHCl₃ Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$

$M_r = 705.44$
 Monoclinic
 $P2_1/c$
 $a = 12.3735 (4) \text{ \AA}$
 $b = 11.4382 (4) \text{ \AA}$
 $c = 22.0706 (11) \text{ \AA}$
 $\beta = 97.480 (3)^\circ$
 $V = 3097.0 (2) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.513 \text{ Mg m}^{-3}$
 $D_m = 1.512 \text{ Mg m}^{-3}$

Data collection

Enraf-Nonius CAD-4 diffractometer
 Profile data from $\theta/2\theta$ scans
 Absorption correction: ψ scans (MolEN; Fair, 1990)
 $T_{\min} = 0.980$, $T_{\max} = 1.000$
 10 936 measured reflections
 7092 independent reflections
 5749 observed reflections
 $[I > 3\sigma(I)]$

Refinement

Refinement on F
 $R = 0.029$
 $wR = 0.036$
 $S = 1.956$
 5397 reflections
 380 parameters
 All parameters refined for H28, other H atoms placed in calculated positions and not refined

Cell parameters from 100 reflections
 $\theta = 12-15^\circ$
 $\mu = 0.966 \text{ mm}^{-1}$
 $T = 294 \text{ K}$
 Parallelepiped with well defined faces
 $0.40 \times 0.30 \times 0.24 \text{ mm}$
 Orange

$R_{\text{int}} = 0.014$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -11 \rightarrow 15$
 $k = -5 \rightarrow 14$
 $l = -27 \rightarrow 28$
 3 standard reflections monitored every 100 reflections
 frequency: 60 min
 intensity decay: 2.9%

$w = 1/\sigma^2(F)$
 where $\sigma(F) = \sigma(F^2)/2F$, $\sigma(F^2) = [\sigma^2(I) + (pF^2)^{1/2}]^{1/2}$ and $p = 0.01$
 $(\Delta/\sigma)_{\text{max}} = 0.01$
 $\Delta\rho_{\text{max}} = 0.635 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.082 \text{ e \AA}^{-3}$
 Extinction correction: none
 Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$) for (A)

	x	y	z	U_{eq}
Ru	0.84808 (1)	-0.13140 (2)	0.39457 (1)	0.03858 (4)
Cl1	0.79535 (5)	-0.17005 (6)	0.49495 (3)	0.0552 (1)
P1	0.70320 (4)	-0.23112 (5)	0.34600 (2)	0.0363 (1)
P2	0.70684 (4)	-0.00298 (5)	0.37383 (2)	0.0373 (1)
C1	0.7143 (2)	-0.2841 (2)	0.26910 (9)	0.0428 (5)
C2	0.7801 (2)	-0.3817 (2)	0.2646 (1)	0.0553 (6)
C3	0.7968 (2)	-0.4246 (3)	0.2080 (1)	0.0699 (8)
C4	0.7488 (3)	-0.3709 (3)	0.1554 (1)	0.0771 (9)
C5	0.6848 (3)	-0.2763 (3)	0.1598 (1)	0.0800 (10)
C6	0.6661 (3)	-0.2324 (2)	0.2161 (1)	0.0616 (8)
C7	0.6360 (2)	-0.3556 (2)	0.3767 (1)	0.0424 (5)
C8	0.5323 (2)	-0.3888 (2)	0.3516 (1)	0.0507 (6)
C9	0.4810 (2)	-0.4826 (2)	0.3754 (1)	0.0643 (8)
C10	0.5344 (3)	-0.5446 (3)	0.4233 (1)	0.0781 (9)
C11	0.6376 (3)	-0.5140 (3)	0.4476 (1)	0.0850 (10)
C12	0.6887 (3)	-0.4185 (2)	0.4255 (1)	0.0641 (8)
C13	0.7089 (2)	0.1092 (2)	0.3146 (1)	0.0439 (5)
C14	0.6703 (2)	0.2220 (2)	0.3209 (1)	0.0595 (8)
C15	0.6747 (3)	0.3040 (3)	0.2748 (1)	0.0743 (9)
C16	0.7167 (3)	0.2754 (3)	0.2229 (1)	0.0775 (9)

C17	0.7546 (3)	0.1643 (3)	0.2158 (1)	0.0746 (9)
C18	0.7513 (2)	0.0812 (2)	0.2613 (1)	0.0569 (6)
C19	0.6507 (2)	0.0744 (2)	0.4345 (1)	0.0431 (5)
C20	0.7237 (2)	0.1189 (3)	0.4814 (1)	0.0602 (8)
C21	0.6871 (3)	0.1831 (3)	0.5284 (1)	0.0726 (9)
C22	0.5774 (3)	0.2002 (2)	0.5282 (1)	0.0676 (8)
C23	0.5038 (2)	0.1546 (2)	0.4827 (1)	0.0610 (6)
C24	0.5404 (2)	0.0922 (2)	0.4355 (1)	0.0505 (6)
C25	0.9739 (2)	−0.0411 (3)	0.3537 (1)	0.0765 (9)
C26	0.9612 (2)	−0.1514 (4)	0.3292 (1)	0.0919 (10)
C27	0.9916 (3)	−0.2333 (3)	0.3752 (2)	0.0987 (11)
C28	1.0212 (2)	−0.1723 (4)	0.4276 (2)	0.0866 (10)
C29	1.0125 (2)	−0.0505 (3)	0.4159 (1)	0.0690 (8)
C30	0.6031 (2)	−0.1104 (2)	0.3417 (1)	0.0434 (5)
C31	0.9577 (4)	−0.1484 (3)	0.0992 (2)	0.095 (1)
C12†	1.0772 (2)	−0.1206 (3)	0.0704 (1)	0.173 (1)
C13‡	0.8677 (3)	−0.0361 (2)	0.0843 (1)	0.153 (1)
C14§	0.9934 (3)	−0.1729 (3)	0.1741 (1)	0.253 (1)
C12'¶	1.0102 (5)	−0.0716 (4)	0.0442 (2)	0.214 (1)
C13'***	0.8799 (4)	−0.0476 (3)	0.1311 (2)	0.195 (1)
C14'††	1.0388 (4)	−0.2139 (4)	0.1509 (3)	0.308 (3)

† Site occupancy 0.639 (3). ‡ Site occupancy 0.573 (3). § Site occupancy 0.667 (3). ¶ Site occupancy 0.335 (3). *** Site occupancy 0.385 (3). †† Site occupancy 0.387 (3).

Table 2. Selected geometric parameters ($\text{\AA}$, $^\circ$) for (A)

Ru—C11	2.4302 (6)	Ru—C29	2.229 (3)
Ru—P1	2.2724 (5)	P1—C1	1.824 (2)
Ru—P2	2.2833 (6)	P1—C7	1.823 (2)
Ru—C25	2.161 (3)	P1—C30	1.849 (2)
Ru—C26	2.149 (3)	P2—C13	1.834 (2)
Ru—C27	2.212 (3)	P2—C19	1.817 (2)
Ru—C28	2.220 (3)	P2—C30	1.851 (2)
C11—Ru—P1	92.85 (2)	Ru—P1—C30	97.57 (7)
C11—Ru—P2	90.60 (2)	Ru—P2—C13	120.75 (8)
P1—Ru—P2	72.07 (2)	Ru—P2—C19	121.46 (7)
Ru—P1—C1	117.08 (8)	Ru—P2—C30	97.14 (7)
Ru—P1—C7	125.92 (7)	P1—C30—P2	92.8 (1)

Compound (B)*Crystal data*

[RuCl(C₅H₅)(C₂₆H₂₄P₂)]·
CHCl₃

$M_r = 719.42$

Monoclinic

$P2_1/c$

$a = 15.592$ (4) $\text{\AA}$

$b = 10.796$ (2) $\text{\AA}$

$c = 19.676$ (6) $\text{\AA}$

$\beta = 109.01$ (1) $^\circ$

$V = 3131$ (1) $\text{\AA}^3$

$Z = 4$

$D_x = 1.526$ Mg m^{−3}

$D_m = 1.515$ Mg m^{−3}

Data collection

Enraf–Nonius CAD-4

diffractometer

Profile data from $\theta/2\theta$ scans

Absorption correction:

ψ scans (MolEN; Fair,
1990)

$T_{\min} = 0.944$, $T_{\max} =$
1.000

8480 measured reflections

7185 independent reflections

5636 observed reflections

$[I > 3\sigma(I)]$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ $\text{\AA}$

Cell parameters from 100
reflections

$\theta = 12\text{--}16^\circ$

$\mu = 0.957$ mm^{−1}

$T = 294$ K

Parallelepiped with well
defined faces

$0.50 \times 0.45 \times 0.38$ mm

Orange

$R_{\text{int}} = 0.009$

$\theta_{\max} = 27.5^\circ$

$h = -7 \rightarrow 19$

$k = -5 \rightarrow 13$

$l = -25 \rightarrow 23$

3 standard reflections

monitored every 100
reflections

frequency: 60 min

intensity decay: 7.3%

Refinement

Refinement on F

$R = 0.031$

$wR = 0.035$

$S = 2.562$

5258 reflections

352 parameters

H-atom parameters not
refined

$w = 1/\sigma^2(F)$

where $\sigma(F) =$

$\sigma(F^2)/2F$, $\sigma(F^2) = [\sigma^2(I)$

$+ (pF^2)^{1/2}$ and $p = 0.01$

$(\Delta/\sigma)_{\max} = 0.01$

$\Delta\rho_{\max} = 0.518$ e $\text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.105$ e $\text{\AA}^{-3}$

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV, Tables

2.2B and 2.3.1)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$) for (B)

	x	y	z	U_{eq}
Ru	0.78207 (1)	0.07158 (2)	0.82429 (1)	0.03713 (5)
C11	0.82931 (5)	0.28483 (6)	0.81371 (4)	0.0543 (3)
P1	0.73868 (4)	0.07735 (6)	0.70250 (3)	0.0382 (1)
P2	0.91250 (4)	−0.01070 (6)	0.81418 (3)	0.0389 (1)
C1	0.6716 (2)	−0.0488 (2)	0.6482 (1)	0.0381 (6)
C2	0.6621 (2)	−0.1606 (3)	0.6792 (1)	0.0471 (8)
C3	0.6119 (2)	−0.2561 (3)	0.6378 (2)	0.0590 (9)
C4	0.5709 (2)	−0.2397 (3)	0.5655 (2)	0.0614 (9)
C5	0.5807 (2)	−0.1309 (3)	0.5335 (2)	0.0594 (9)
C6	0.6306 (2)	−0.0355 (3)	0.5747 (1)	0.0502 (8)
C7	0.6711 (2)	0.2101 (3)	0.6574 (1)	0.0476 (8)
C8	0.6951 (2)	0.2905 (3)	0.6128 (2)	0.073 (1)
C9	0.6380 (3)	0.3884 (3)	0.5809 (2)	0.091 (1)
C10	0.5596 (3)	0.4065 (3)	0.5936 (2)	0.090 (1)
C11	0.5348 (3)	0.3271 (4)	0.6376 (2)	0.092 (1)
C12	0.5899 (2)	0.2287 (3)	0.6696 (2)	0.074 (1)
C13	0.9429 (2)	−0.1664 (2)	0.8515 (1)	0.0437 (6)
C14	1.0093 (2)	−0.1848 (3)	0.9173 (2)	0.0590 (9)
C15	1.0286 (2)	−0.3012 (3)	0.9470 (2)	0.070 (1)
C16	0.9811 (2)	−0.4026 (3)	0.9111 (2)	0.0678 (9)
C17	0.9148 (2)	−0.3861 (3)	0.8461 (2)	0.0650 (9)
C18	0.8960 (2)	−0.2697 (3)	0.8169 (2)	0.0557 (9)
C19	1.0203 (2)	0.0712 (2)	0.8531 (1)	0.0438 (6)
C20	1.0268 (2)	0.1655 (3)	0.9021 (2)	0.0532 (9)
C21	1.1084 (2)	0.2273 (3)	0.9327 (2)	0.066 (1)
C22	1.1824 (2)	0.1967 (3)	0.9138 (2)	0.069 (1)
C23	1.1769 (2)	0.1029 (3)	0.8657 (2)	0.068 (1)
C24	1.0966 (2)	0.0391 (3)	0.8355 (2)	0.0561 (9)
C25	0.7423 (2)	−0.0814 (3)	0.8790 (2)	0.0614 (9)
C26	0.6641 (2)	−0.0187 (3)	0.8377 (2)	0.0616 (9)
C27	0.6662 (2)	0.1016 (3)	0.8651 (2)	0.0674 (9)
C28	0.7462 (2)	0.1122 (3)	0.9227 (2)	0.075 (1)
C29	0.7938 (2)	0.0009 (4)	0.9327 (2)	0.069 (1)
C30	0.8408 (2)	0.0755 (3)	0.6758 (1)	0.0484 (8)
C31	0.9014 (2)	−0.0278 (3)	0.7182 (1)	0.0484 (8)
C32	0.6769 (2)	0.4695 (3)	0.8590 (2)	0.077 (1)
C12	0.57701 (7)	0.4138 (1)	0.86726 (8)	0.1270 (4)
C13	0.75163 (9)	0.5108 (2)	0.94169 (7)	0.1451 (6)
C14	0.6557 (1)	0.5940 (1)	0.80141 (8)	0.1568 (6)

Table 4. Selected geometric parameters ($\text{\AA}$, $^\circ$) for (B)

Ru—C11	2.4466 (7)	P1—C1	1.833 (2)
Ru—P1	2.2688 (7)	P1—C7	1.828 (3)
Ru—P2	2.2863 (7)	P1—C30	1.832 (3)
Ru—C25	2.169 (3)	P2—C13	1.834 (3)
Ru—C26	2.173 (3)	P2—C19	1.832 (2)
Ru—C27	2.227 (4)	P2—C31	1.849 (3)
Ru—C28	2.226 (4)	C30—C31	1.523 (4)
Ru—C29	2.217 (3)		

Cl1—Ru—P1	83.04 (3)	Ru—P2—Cl3	116.3 (1)
Cl1—Ru—P2	93.28 (3)	Ru—P2—C19	119.82 (9)
P1—Ru—P2	83.48 (2)	Ru—P2—C31	109.73 (8)
Ru—P1—C1	120.81 (9)	P1—C30—C31	107.1 (2)
Ru—P1—C7	117.42 (9)	P2—C31—C30	108.7 (2)
Ru—P1—C30	108.33 (8)		

Both crystals were mounted in random orientations on glass fibers. Rotation photographs were used to locate reflections for subsequent indexing. Axial photographs confirmed the axial lengths for both unit cells and $2/m$ Laue diffraction symmetry. The reflection conditions $h0l$ ($l = 2n$) and $0k0$ ($k = 2n$) resulted in the space-group assignment as $P2_1/c$. Data for both compounds included a number of redundant data at low angle as a measure of the precision and quality of the data sets. Both structures were solved *via* direct methods which resulted in the Ru-atom position and the positions of a small group of atoms including Cl and P. The structures were completed by running full-matrix least-squares cycles on the partial model with isotropic displacement parameters followed by difference Fourier syntheses.

Large positive residuals [greater than $1.0 \text{ e } \text{\AA}^{-3}$ for (A) and greater than $3.0 \text{ e } \text{\AA}^{-3}$ for (B)] remained in the difference Fourier maps for both structures after the assignment of all non-H atoms in the compounds. Examination of these residuals revealed the presence of a chloroform molecule in the asymmetric unit of both structures. Disorder of the chloroform molecule in the structure of (A) required modeling with two sets of Cl atoms with fractional site occupancies. The sums of the occupancies for corresponding Cl atoms ranged from 0.958 to 1.054. Simultaneous refinement of both sets of Cl atoms was ill-behaved, so one set of Cl atoms was refined with strongly damped shifts while the parameters for the other set were fixed. The roles of both sets of Cl atoms were alternated until the refinement converged.

All non-H atoms were refined with anisotropic displacement parameters. H atoms were located in the difference Fourier maps but were placed at idealized positions; each assigned an isotropic displacement parameter 30% larger than the isotropic displacement parameter of the C atom to which it is bonded. The only exception to this procedure was for H28 on the disordered chloroform molecule in the structure of (A). This single H atom was located in difference Fourier maps and its position and isotropic displacement parameter refined assuming full occupancy. No other residual Fourier peak could be assigned to a second H atom of the disordered molecule. H-atom positions were updated throughout the final cycles of refinement. Examination of strong, low-angle reflections revealed no systematic extinction effects.

For both compounds, data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structures: *MULTAN11/82* (Main *et al.*, 1982); program(s) used to refine structures: *MolEN*; molecular graphics: *ORTEPII* (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms and H28 for (A), and bond distances and angles involving non-H atoms for (B) have been deposited with the IUCr (Reference: BK1121). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tricarbonyl(η^5 -2,4-dimethylpentadienyl)-iron Tetrafluoroborate

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Abstract

The title compound, [Fe(C₇H₁₁)(CO)₃]BF₄, has been found to have the expected piano-stool geometry, except that the unique carbonyl ligand is tilted towards the

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