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The Crystal and Molecular Structure of Dicarbonylbis(η-cyclopentadienyl)-μ-(2,3-η-1,1,1,4,4,4-hexafluoro-2-butenyl)-dirhodium

BY RON S. DICKSON, SHARANN H. JOHNSON, HOWARD P. KIRSCH AND DOUGLAS J. LLOYD

Department of Chemistry, Monash University, Wellington Road, Clayton, Victoria, Australia 3168

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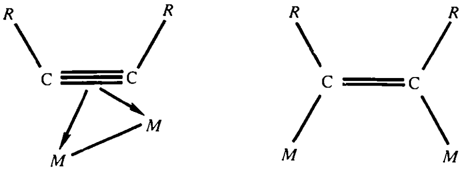
The structure of $(\eta\text{-}C_5H_5)_2Rh_2(CO)_2(CF_3C_2CF_3)$ has been determined from 3893 diffractometer data and refined by least squares to $R = 0{\cdot}075$. The compound crystallizes in the triclinic space group $P\bar{1}$ with $a = 9{\cdot}322\ (5)$, $b = 12{\cdot}139\ (6)$, $c = 8{\cdot}671\ (5)$ Å, $\alpha = 103{\cdot}60\ (5)$, $\beta = 68{\cdot}98\ (5)$, $\gamma = 109{\cdot}87\ (5)°$, $Z = 2$. The two Rh atoms are essentially coplanar with the four C atoms of the $CF_3C_2CF_3$ ligand; the Rh—C distances are 2·031 (10) and 2·054 (10) Å. This geometry is consistent with attachment of the ligand to the Rh atoms by two σ bonds. The ligand is *cis* bent, and the distance between the ethenyl C atoms is 1·269 (14) Å. The distance of 2·682 (1) Å between the two Rh atoms is consistent with a Rh—Rh bonding interaction. One CO group is attached to each Rh and the Rh—C—O bonds are approximately normal to the Rh_2C_4 plane. Within each molecule, the two CO groups, and consequently the two $(\eta\text{-}C_5H_5)$ groups, assume a *trans* arrangement.

Introduction

Alkyne–transition-metal complexes are important intermediates in synthesis. Complexes in which the alkyne adopts a μ-bridging position between two metal atoms are of particular interest. The structures of several of these complexes have been determined by X-ray diffraction, and a representation of the main structural features is shown in Fig. 1(*a*). The alkyne is positioned normal to and above the metal–metal bond axis. The C–C distance is longer in the coordinated alkyne than in the free alkyne, and the substituents assume a *cis* bent geometry. The Co complexes $Co_2(CO)_6(RC_2R)$ (Dickson & Fraser, 1974, Table VI) and the Ni complexes $(\eta\text{-}C_5H_5)_2Ni_2(RC_2R)$ (Mills & Shaw, 1968) are typical of this class of compound.

Studies of the structures of several hexafluorobut-2-yne–dimetal complexes have revealed a different bonding mode. $(Ph_3P)_2Au_2(CF_3C_2CF_3)$ (Gilmore & Woodward, 1971), $Fe_2(CO)_6(SCF_3)_2(CF_3C_2CF_3)$ (Davidson, Harrison, Sharp & Sim, 1972) and $(Ph_3P)_2Ir_2(NO)_2(CF_3C_2CF_3)$ (Clemens, Green, Kuo,



(*a*) (*b*)

Fig. 1. Representations of bonding models for μ-alkyne–dimetal complexes.

Fritchie, Mague & Stone, 1972) incorporate an alkyne unit which is σ-bonded to and coplanar with the two metal atoms [Fig. 1(*b*)]. In these complexes, the metal–metal distance is too long to be consistent with a metal–metal bonding interaction.

An alkyne–dimetal complex of formula $(\eta\text{-}C_5H_5)_2\text{-}Rh_2(CO)_2(CF_3C_2CF_3)$ was isolated from the reaction between $(\eta\text{-}C_5H_5)Rh(CO)_2$ and hexafluorobut-2-yne (Dickson & Kirsch, 1972). A study of the structure of this complex has revealed that the alkyne unit is σ-bonded to the two Rh atoms, and that there is a bonding interaction between the two Rh atoms (Dickson, Kirsch & Lloyd, 1975). We now report the details of the structure analysis.

Experimental

Orange platelets (Dickson & Kirsch, 1972) were obtained from hexane. Crystals of more uniform dimensions were obtained from *n*-amyl acetate. A crystal 0·18 × 0·25 × 0·18 mm was mounted on a quartz fibre.

Crystal data

$C_{16}H_{10}Rh_2F_6O_2$, $M_r = 554{\cdot}04$, triclinic, $P\bar{1}$, $a = 9{\cdot}322\,(5)$, $b = 12{\cdot}139\,(6)$, $c = 8{\cdot}671\,(5)$ Å, $\alpha = 103{\cdot}60\,(5)$, $\beta = 68{\cdot}98\,(5)$, $\gamma = 109{\cdot}87\,(5)°$, $U = 854{\cdot}17$ Å^3. $D_m = 2{\cdot}2$ (by flotation in chloroform and 1,1,2,2-tetrabromoethane), $Z = 2$, $D_c = 2{\cdot}15$ g cm^{-3}, $F(000) = 531{\cdot}99$, $\mu = 19{\cdot}5$ cm^{-1} for Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7107$ Å). Cell parameters were determined from 25 reflexions and were calculated by the standard Philips program.

Intensity measurements

Intensities were collected on a Philips PW 1100 diffractometer with Mo $K\alpha$ radiation. A unique data set was collected out to $2\theta = 60°$ by the ω scan technique at a scan rate of 0·04° s^{-1}, and over a range about the calculated scattering position given by $\pm(1{\cdot}4 + 0{\cdot}3\tan\theta)/2°$. 4855 independent reflexions were

Table 1. *Final positional parameters* (×10^4) *and thermal parameters* (×10^4)

Estimated standard deviations are in parentheses.

	x	y	z	B (Å^2)
Rh(1)	1780 (1)	3209 (1)	3042 (1)	†
Rh(2)	4234 (1)	2291 (1)	1337 (1)	†
C(1)	1410 (21)	2455 (16)	6397 (21)	804 (41)
C(2)	2259 (12)	2403 (9)	4562 (13)	445 (21)
C(3)	3365 (11)	1919 (8)	3722 (12)	401 (19)
C(4)	4100 (18)	1181 (13)	4296 (19)	665 (33)
C(5)	3274 (12)	4535 (9)	3609 (13)	424 (20)
C(6)	2766 (14)	958 (10)	798 (14)	502 (24)
C(7)	336 (13)	2345 (10)	1210 (14)	485 (23)
C(8)	−522 (16)	1909 (11)	2759 (16)	588 (28)
C(9)	−832 (19)	2927 (14)	3943 (20)	763 (38)
C(10)	−175 (18)	3904 (13)	3117 (19)	710 (35)
C(11)	592 (14)	3572 (10)	1444 (15)	537 (26)
C(12)	5960 (18)	3934 (13)	280 (18)	703 (35)
C(13)	6581 (17)	3611 (12)	1339 (17)	656 (32)
C(14)	6835 (19)	2517 (14)	662 (20)	750 (38)
C(15)	6463 (20)	2128 (15)	−801 (21)	819 (42)
C(16)	5855 (21)	3014 (16)	−1024 (22)	857 (44)
O(1)	4182 (12)	5385 (9)	3992 (13)	699 (24)
O(2)	1882 (15)	111 (11)	412 (16)	954 (34)
F(1)	912 (16)	3395 (13)	7061 (18)	1388 (44)
F(2)	2265 (21)	2530 (14)	7288 (21)	1691 (58)
F(3)	105 (19)	1631 (13)	6707 (19)	1550 (53)
F(4)	4804 (16)	475 (11)	3066 (16)	1248 (39)
F(5)	5166 (12)	1791 (9)	4989 (12)	933 (28)
F(6)	3064 (20)	416 (14)	5181 (21)	1646 (56)

† Anisotropic thermal parameters (×10^4) were used for the Rh atoms. The temperature factor is of the form

$$\exp[-2\pi^2(U_{11}h^2a^{*2} + \cdots + 2U_{23}b^*c^*kl\cdots)].$$

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rh(1)	288 (3)	488 (4)	312 (3)	11 (3)	−102 (3)	113 (3)
Rh(2)	335 (4)	380 (4)	395 (4)	18 (3)	−144 (3)	120 (3)

Table 2. *Intramolecular bonding distances* (Å) *and selected non-bonding distances* (Å)

(*a*) Bonding distances

Rh(1)–Rh(2)	2·682 (1)	C(1)–F(1)	1·307 (19)
Rh(1)–C(2)	2·054 (10)	C(1)–F(2)	1·265 (20)
Rh(2)–C(3)	2·031 (10)	C(1)–F(3)	1·271 (20)
Rh(1)–C(5)	1·835 (10)	C(4)–F(4)	1·325 (17)
Rh(2)–C(6)	1·818 (11)	C(4)–F(5)	1·271 (16)
Rh(1)–C(7)	2·286 (11)	C(4)–F(6)	1·256 (20)
Rh(1)–C(8)	2·247 (13)	C(5)–O(1)	1·158 (13)
Rh(1)–C(9)	2·202 (16)	C(6)–O(2)	1·144 (16)
Rh(1)–C(10)	2·230 (15)	C(7)–C(8)	1·414 (16)
Rh(1)–C(11)	2·249 (12)	C(8)–C(9)	1·449 (19)
Rh(2)–C(12)	2·243 (15)	C(9)–C(10)	1·373 (21)
Rh(2)–C(13)	2·231 (14)	C(10)–C(11)	1·398 (18)
Rh(2)–C(14)	2·214 (16)	C(11)–C(7)	1·402 (15)
Rh(2)–C(15)	2·266 (17)	C(12)–C(13)	1·428 (19)
Rh(2)–C(16)	2·229 (17)	C(13)–C(14)	1·383 (20)
C(2)–C(3)	1·269 (14)	C(14)–C(15)	1·369 (22)
C(1)–C(2)	1·498 (20)	C(15)–C(16)	1·451 (23)
C(3)–C(4)	1·546 (17)	C(16)–C(12)	1·395 (21)

(*b*) Non-bonding distances

Rh(1)···C(1)	3·120 (17)	Rh(2)···C(4)	3·113 (15)
Rh(1)···C(3)	2·759 (10)	Rh(2)···C(2)	2·743 (11)

measured; 3893 obeyed the condition $F_o^2 > 3\sigma(F_o^2)$ and were used in the subsequent calculations. Three standard reflexions, monitored every 2·0 h, showed no significant variation.

The data were processed with a program written for the PW 1100 diffractometer (Hornstra & Stubbe, 1972). A value of 0·04 was used for p in the estimation of $\sigma(F_o^2)$. All reflexions were corrected for Lorentz and polarization effects, but not for extinction or absorption.

Structure determination and refinement

Scattering factors for Rh, C, F and O were taken from Cromer & Waber (1965). The program used for refinement was *SHELX* (Sheldrick, 1975). The diagrams were drawn with *ORTEP* (Johnson, 1965). All major calculations were performed on the Monash University Burroughs 6700 computer.

Solution of the structure was achieved by Patterson and difference Fourier methods. In the least-squares calculations the function $\Sigma\, \omega(|F_o| - |F_c|)^2$ was minimized; the weight, ω, was taken as k/σ^2, where k is a constant determined for each least-squares refinement. The reflexions 110 and 020 were not included in the final cycle because they were affected by extinction. Refinement of the structure led to $R_1 = 0{\cdot}075$ and $R_2 = 0{\cdot}085$, where $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ and $R_2 = [\Sigma\, \omega^{1/2}(|F_o| - |F_c|)/\Sigma\, \omega^{1/2}|F_o|]$. The smooth refinement and final model confirmed that the space group $P\bar{1}$ was correct. H atoms were sought in their ideal locations in a difference synthesis. Although some were located, others were not significantly above residual maxima and we chose to omit all H atoms from the determination.

The positional and thermal parameters are given in Table 1 together with the associated standard deviations estimated from the inverse matrix. Bond lengths are in Table 2 and bond angles in Table 3.*

Results and discussion

The crystal structure is shown in Fig. 2 and consists of the packing of discrete molecules. There are no unusually short intermolecular contacts. Views of the molecule are presented in Figs. 3 and 4. Fig. 3 also indicates the labelling of the atoms.

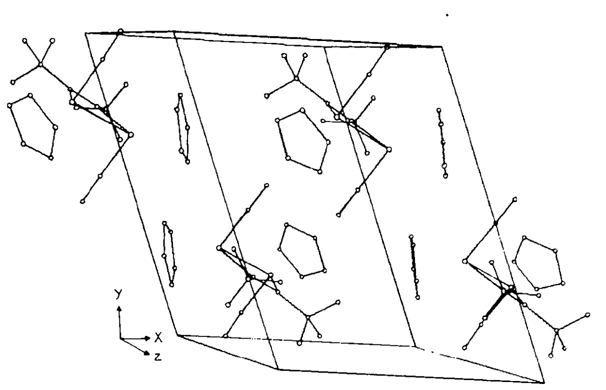


Fig. 2. The molecular packing in the unit cell. The view is along the Z axis.

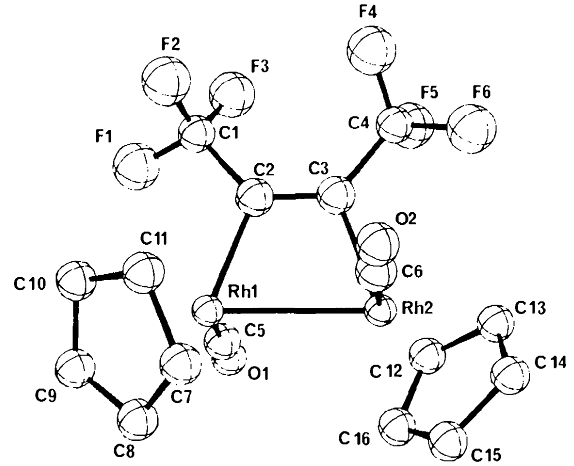


Fig. 3. A drawing of $(\eta\text{-}C_5H_5)_2Rh_2(CO)_2(CF_3C_2CF_3)$, with H atoms omitted. The 50% probability ellipsoids are shown.

Table 3. *Selected bond angles* (°)

Rh(1)–Rh(2)–C(3)	70·1 (0·3)	C(5)–Rh(1)–Rh(2)	86·6 (0·3)	F(4)–C(4)–F(6)	98·9 (1·4)
Rh(2)–Rh(1)–C(2)	69·4 (0·3)	C(6)–Rh(2)–Rh(1)	87·2 (0·4)	F(5)–C(4)–F(6)	110·8 (1·5)
Rh(1)–C(2)–C(3)	110·0 (0·8)	C(2)–C(1)–F(1)	114·3 (1·4)	C(7)–C(8)–C(9)	105·4 (1·1)
Rh(2)–C(3)–C(2)	110·3 (0·8)	C(2)–C(1)–F(2)	115·0 (1·6)	C(8)–C(9)–C(10)	108·6 (1·4)
C(1)–C(2)–C(3)	127·8 (1·1)	C(2)–C(1)–F(3)	111·1 (1·5)	C(9)–C(10)–C(11)	109·1 (1·3)
C(2)–C(3)–C(4)	129·2 (1·0)	C(3)–C(4)–F(4)	113·5 (1·2)	C(10)–C(11)–C(7)	107·8 (1·1)
Rh(1)–C(2)–C(1)	122·1 (0·9)	C(3)–C(4)–F(5)	114·4 (1·2)	C(11)–C(7)–C(8)	109·0 (1·0)
Rh(2)–C(3)–C(4)	120·4 (0·8)	C(3)–C(4)–F(6)	113·1 (1·3)	C(12)–C(13)–C(14)	107·7 (1·3)
Rh(1)–C(5)–O(1)	178·1 (0·9)	F(1)–C(1)–F(2)	100·4 (1·6)	C(13)–C(14)–C(15)	110·9 (1·5)
Rh(2)–C(6)–O(2)	177·7 (1·1)	F(1)–C(1)–F(3)	101·8 (1·6)	C(14)–C(15)–C(16)	106·1 (1·5)
C(2)–Rh(1)–C(5)	85·9 (0·4)	F(2)–C(1)–F(3)	118·0 (1·8)	C(15)–C(16)–C(12)	108·3 (1·5)
C(3)–Rh(2)–C(6)	88·9 (0·4)	F(4)–C(4)–F(5)	104·8 (1·3)	C(16)–C(12)–C(13)	106·9 (1·3)

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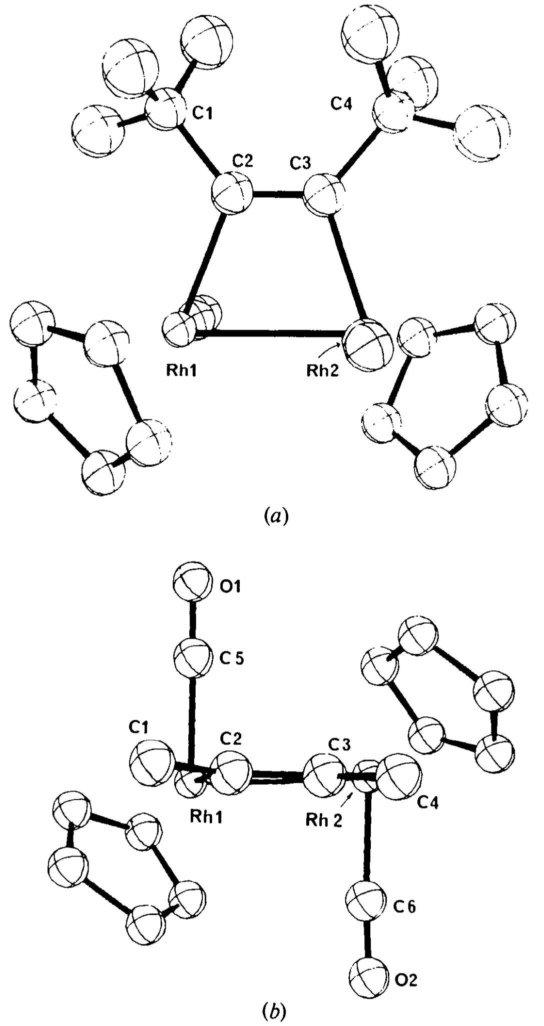
* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32386 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

The two Rh atoms are essentially coplanar with the four C atoms of the $CF_3C_2CF_3$ ligand. The perpendicular displacements from the plane through these atoms are indicated in Table 4 and torsion angles are shown in Fig. 5. The distance of 2·682 (1) Å between the two Rh atoms is consistent with a normal Rh—Rh bonding interaction and is identical to the distance in (η-$C_5H_5)_2Rh_2(CO)_3$ (Mills & Nice, 1967).

One CO group is attached to each Rh atom, and the two Rh—CO bonds are approximately normal to the Rh_2C_4 plane. Appropriate torsion angles are shown in Fig. 5. The two CO groups within each molecule assume a *trans* arrangement. This differs from our earlier proposal of a *cis* orientation, which was based on an interpretation of the infrared spectrum (Dickson & Kirsch, 1972). The ^{13}C NMR spectra of solutions of (η-$C_5H_5)_2Rh_2(CO)_2(CF_3C_2CF_3)$ indicate that the CO groups scramble between bridging and terminal positions. The relationship between this fluxional behaviour in solution and the solid-state structure has been discussed (Todd, Wilkinson, Rausch, Gardner & Dickson, 1975). The average Rh—C(carbonyl) and C—O distances are 1·82 (1) and 1·15 (1) Å respectively, and each Rh—C—O fragment is essentially linear. These distances are very similar to the corresponding distances in (η-$C_5H_5)_2Rh_2(CO)_3$ (Mills & Nice, 1967).

The attachment of the $CF_3C_2CF_3$ ligand to the Rh atoms by two σ bonds gives a four-membered ring. With the exception of the benzyne—diiron complex $Fe_2(CO)_8(C_6F_4)$, which is not strictly an alkyne complex, there are no confirmed examples of alkyne—dimetal complexes which incorporate both a metal—metal bond and a σ-bonding arrangement like that



(*a*)

(*b*)

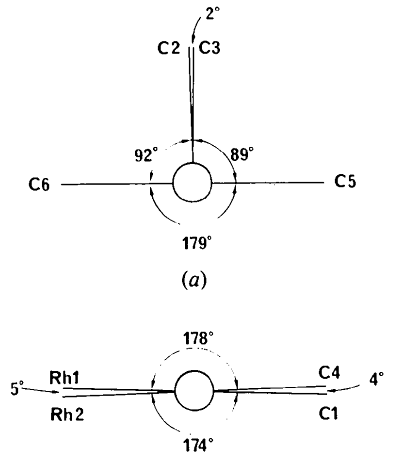
Fig. 4. Two views of the molecule showing (*a*) the *trans* arrangement of the carbonyls and (*b*) the planarity of the Rh_2—C_4 fragment.

Table 4. *Equations of the mean planes and deviations* (Å) *of atoms from these planes*

Each plane is represented by an equation of the type $Ax + By + Cz - D = 0$, with x,y,z in triclinic coordinates.

Plane 1: Rh(1), Rh(2), C(1), C(2), C(3), C(4)
$-0{\cdot}5321x - 0{\cdot}8191y - 0{\cdot}2145z + 4{\cdot}0040 = 0$
Plane 2: C(7), C(8), C(9), C(10), C(11)
$-0{\cdot}9711x - 0{\cdot}1341y - 0{\cdot}1973z + 0{\cdot}2789 = 0$
Plane 3: C(12), C(13), C(14), C(15), C(16)
$0{\cdot}6692x + 0{\cdot}5283y - 0{\cdot}5226z - 4{\cdot}9243 = 0$

Plane 1		Plane 2		Plane 3	
Rh(1)	0·058	C(7)	0·015	C(12)	0·006
Rh(2)	−0·073	C(8)	−0·007	C(13)	0·005
C(1)	0·014	C(9)	−0·003	C(14)	−0·014
C(2)	0·023	C(10)	0·013	C(15)	0·017
C(3)	−0·065	C(11)	−0·017	C(16)	−0·013
C(4)	0·042	Rh(1)	−1·896	Rh(2)	−1·890



(*a*)

(*b*)

Fig. 5. The torsion angles (°) about (*a*) the Rh(1)—Rh(2) and (*b*) the C(2)—C(3) bonds.

shown in Fig. 1(*b*). The Rh—C distances of 2·04 (1) Å are similar to Rh—C(sp^2) distances observed for other molecules (Churchill, 1970; Mague, 1970; Ricci & Ibers, 1971).

The $CF_3C_2CF_3$ ligand is *cis* bent with C=C—CF_3 angles of approximately 130°; the C=C—Rh and Rh—C—CF_3 angles are about 110 and 120° respectively. Deviations from the normal olefinic angle of 120° are presumably a result of constraints imposed by the Rh—Rh and Rh—C distances.

The distance between the ethenyl C atoms is 1·269 (14) Å. This is intermediate between the normal C—C triple-bond (1·20 Å) and double-bond (1·34 Å) distances, and it is not easy to understand why the bond in the coordinated ligand is so short. A similar distance of 1·27 (3) Å has been reported for $Ir_2(PPh_3)_2(NO)_2(CF_3C_2CF_3)$ (Clemens *et al.*, 1972), but a normal C=C distance of 1·34 (1) Å is observed for $Fe_2(CO)_6(SCF_3)_2(CF_3C_2CF_3)$ (Davidson *et al.*, 1972). The cyclopentadienyl C atoms are coplanar (Table 4) and the geometry within the η^5-cyclopentadienyl rings is regular within the accuracy of our determination. The Rh to cyclopentadienyl plane distances are 1·90 and 1·89 Å, very similar to those found in other (η-C_5H_5)Rh complexes.

Recent investigations have led to the isolation of several other complexes of general formula (η-$C_5H_5)_2M_2(CO)_2(RC_2R)$. Thus, the complexes with M = Rh, RC_2R = $C_6F_5C_2C_6F_5$ (Rausch & Gardner, 1973); M = Ir, RC_2R = $C_6F_5C_2C_6F_5$ (Gardner, Andrews & Rausch, 1973); and M = Ir, RC_2R = $CF_3C_2CF_3$ (Dickson & Corrigan, 1976) have all been characterized spectroscopically and all appear to be structurally analogous. This has been confirmed for the decafluorodiphenylacetylene–dirhodium complex by an X-ray crystallographic investigation (Dahl & Broach, 1973). It is likely, therefore, that such complexes are common products from reactions between alkynes and (η-$C_5H_5)M(CO)_2$ compounds.

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