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VINYLIDENE COMPLEXES OF TRANSITION METALS

III *. DERIVATIVES OF CYCLOPENTADIENYLTRICARBONYL RHENIUM WITH PHENYLVINYLIDENE LIGANDS. CRYSTAL AND MOLECULAR STRUCTURE OF $Cp(CO)_2Re[C{=}C(Ph){-}C(Ph){=}CH_2]Re(CO)_2Cp$

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Summary

The novel complexes $CpRe(C{=}CHPh)(CO)_2$ and $Cp_2Re_2(\mu\text{-}C{=}CHPh)(CO)_4$ containing a terminal and a bridging phenylvinylidene ligand respectively and the binuclear complex $Cp(CO)_2Re[C{=}C(Ph)C(Ph){=}CH_2]Re(CO)_2Cp$ were obtained in the reaction of $CpRe(CO)_3$ with $PhC{\equiv}CH$.

According to an X-ray study of the latter complex the unusual bridging ligand is η^1-bonded to one Re atom and η^2-bonded to the other.

Introduction

Several types of transition metal compounds with organic η^1-ligands are known. Along with σ-organic and carbene complexes where the bond order of the metal—carbon bond is 1—1.2 [2], compounds containing double and triple metal—carbon bonds are being extensively studied at present [1,3]. The synthesis and properties of the rhenium complexes $CpRe(C{=}CHPh)(CO)_2$ and $Cp(CO)_2Re[C{=}C(Ph){-}C(Ph){=}CH_2]Re(CO)_2Cp$ with double metal—carbon bonds and also of the complex $Cp_2Re_2(\mu\text{-}C{=}CHPh)(CO)_4$ which were obtained in the reaction of $CpRe(CO)_3$ with $PhC{\equiv}CH$ are reported for the first time in the present paper.

Results and discussion

Previously we have shown that phenylacetylene π-bonded to the Mn atom in $CpMn(CO)_2(PhC{\equiv}CH)$ undergoes a rearrangement into a carbene type phenyl-

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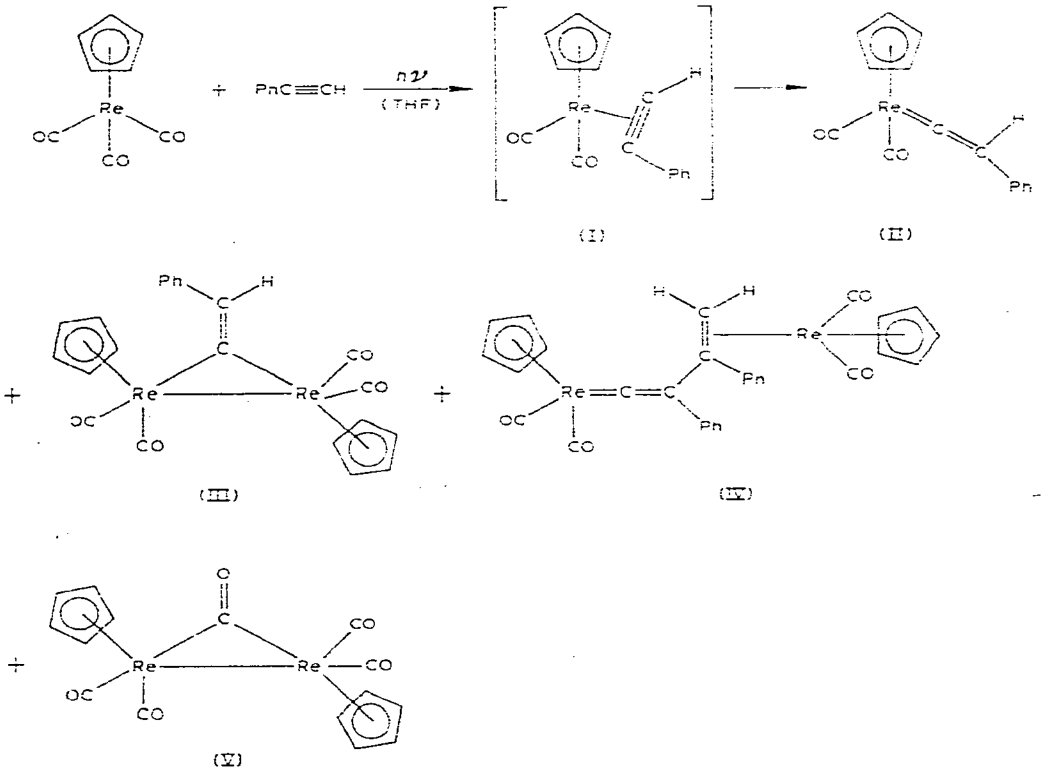
* For part II see ref. 1.

vinylidene ligand. Carbene (>C=CHPh) which is unstable in the free state is stabilized by coordination with a Mn atom forming stable $CpMn(C{=}CHPh)(CO)_2$ and $Cp_2Mn_2(\mu\text{-}C{=}CHPh)(CO)_4$ complexes [1].

In order to determine the influence of a transition metal on the capacity of π-coordinated phenylacetylene to rearrange into a phenylvinylidene ligand we studied the reaction of phenylacetylene with π-cyclopentadienyl rhenium tricarbonyl.

Several complexes were obtained by chromatography of the products of the photochemical reaction between $CpRe(CO)_3$ and $PhC{\equiv}CH$ in THF (see Scheme 1).

SCHEME 1



Some physical properties of compounds II—IV are given in Table 1.

The unstable π-complex I was not isolated but its formation in the first stage of the reaction was confirmed by IR spectroscopy in the 2200—1600 cm^{-1} range. In addition to ν(CO) bands of $CpRe(CO)_3$ and II the spectrum contains a ν(C≡C) band at 1840 cm^{-1} and two ν(CO) bands at 1980 and 1911 cm^{-1} (cyclohexane solutions). The two latter bands are close to those of $CpRe(CO)_2(PhC{\equiv}CPh)$ (1982 and 1911 cm^{-1} in cyclohexane).

The π-acetylenic complex I undergoes rearrangement into the stable complex II. The yield of II depends on the reaction conditions and decreases with a temperature increase. In contrast to $CpMn(C{=}CHPh)(CO)_2$ which is formed in a basic medium (on alumina at pH 9—10), II was isolated on SiO_2 in neutral conditions.

TABLE 1

PHYSICAL PROPERTIES OF RHENIUM VINYLIDENE COMPLEXES

Compound	Colour	M.p. (°C)	IR ν(CO) (cm^{-1}) (in cyclohexane)
$CpRe(C{=}CHPh)(CO)_2$ (II)	red	75—76	2003, 1938
$Cp_2Re_2(\mu\text{-}C{=}CHPh)(CO)_4$ (III)	yellow	193—194	1983, 1953, 1916
$Cp_2Re_2[C{=}C(Ph){-}C(Ph){=}CH_2](CO)_4$ (IV)	light red	150—152	2000, 1982, 1930, 1914

The red crystalline complex II is readily soluble in organic solvents, sublimates without decomposition at 80—100°C/1 × 10^{-2} mmHg. Its IR spectrum has two bands in the region of carbonyl stretching frequencies (Table 1).

Comparison of the ν(CO) frequencies of $CpRe(C{=}CHPh)(CO)_2$ with published data on $CpRe(CO)_2L$ complexes [4] indicates a strong electron-acceptor capacity of the phenylvinylidene ligand. The Raman spectrum of II has an intensive line at 1594 cm^{-1}, which we assigned to the ν(C=C) of the vinylidene ligand. In the IR spectrum ν(C=C) is at 1591 cm^{-1}.

According to a preliminary X-ray study of II * the vinylidene carbon forms a double bond with the metal atom and the Re=C=C fragment represents a linear metalallene system.

The data of the ^{13}C NMR spectrum of II, given in Table 2, are consistent with the structure of this compound. The C^1 atom of the vinylidene ligand reveals a single signal with a strong downfield shift.

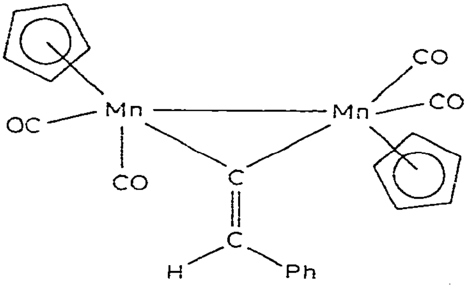
It is interesting that unlike $CpMn(C{=}CHPh)(CO)_2$ which gradually converts into $Cp_2Mn_2(\mu\text{-}C{=}CHPh)(CO)_4$ with a bridging phenylvinylidene ligand [1], II is quite stable and does not convert into III under normal conditions.

Formation of III in the reaction of $CpRe(CO)_3$ with phenylacetylene is apparently due to interaction of the complex II already formed with coordinatively and electronically deficient $[CpRe(CO)_2]$ present in the reaction mixture.

Complex III can be obtained directly from II by treating it with a water/alcohol/base mixture

$$\underset{\text{(II)}}{CpRe(C{=}CHPh)(CO)_2} \xrightarrow{\text{KOH/MeOH}} \underset{\text{(III)}}{Cp_2Re_2(\mu\text{-}C{=}CHPh)(CO)_4}$$

Complex III is a yellow crystalline substance readily soluble in polar organic solvents. It is stable under normal conditions and sublimates without decomposition at 80—100°C/1 × 10^{-2} mmHg. Its IR spectrum contains three ν(CO) bands (Table 1) whose positions and intensities are similar to those in the spectrum of



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* This study was carried out by V.G. Andrianov and Yu.T. Struchkov.

TABLE 2

^{13}C NMR DATA FOR $CpRe(C{=}CHPh)(CO)_2$ [a]

C_5H_5	CO	$C^1{=}C^2{<}^{C_6H_5}_{H}$					
		C^1	C^2	C_6H_5			
				C_{key}	C_{ortho}	C_{meta}	C_{para}
90.37	198.55	329.49	119.49	128.66	125.53	124.88	129.52
(180.7)			(155.6)		(160.0)	(158.7)	(158.7)

[a] $\delta(^{13}C)$ (ppm), in relation to TMS; The $^1J(^{13}C—^1H)$ coupling constants (Hz) in parantheses.

[1] where a *trans*-arrangement of Cp rings in relation to the Mn—Mn bond was established by X-ray study [5]. The ν(C=C) frequency of the bridging vinylidene ligand in III is at 1555 cm^{-1} both in the Raman and in the IR spectra.

The mass spectrum of III has an intensive peak at $P^+ = 716$ m/e corresponding to a molecular ion and peaks of $[P - nCO]^+$ ions (n = 1—4). Elimination of the C=CHPh ligand from a completely decarbonylated ion indicates that the metal—phenylvinylidene bond is stronger than the Re—CO bond.

In contrast to II and III both formed in the photochemical reactions of $CpRe(CO)_3$ with phenylacetylene and in the reaction of the latter with intermediate $CpRe(CO)_2(THF)$, IV is produced only by UV irradiation of a mixture of $CpRe(CO)_3$ with phenylacetylene in THF. IV is readily soluble in polar organic solvents and is stable under normal conditions. Its IR spectrum contains four bands in the ν(CO) region (Table 1). The mass spectrum reveals a peak at $P^+ = 818$ m/e.

The structure of IV was determined by an X-ray study and turned out to be quite unexpected. The molecule of IV contains an unusual bridging 2,3-diphenylbutadienylidene $[C{=}C(Ph){-}C(Ph){=}CH_2]$ ligand. Presumably formation of IV involves II, phenylacetylene and $[CpRe(CO)_2]$ species and requires UV irradiation.

Light red crystals of $Cp(CO)_2Re[C{=}C(Ph){-}C(Ph){=}CH_2]Re(CO)_2Cp$ are monoclinic: $a = 16.91(1)$. $b = 8.206(4)$, $c = 18.99(1)$ Å, $\beta = 106.66(4)°$, $V = 2524(2)$ Å^3, Mol. wt. 818.9, $D_{obs}^{20°}$ 2.09 ± 0.03, D_{calc} 2.152 g cm^{-3} (at −120°C), $Z = 4$, space group $P2_1/n$. Intensities of 3030 independent reflections were measured at −120°C with a Syntex $P2_1$ diffractometer with a low-temperature LT-1 attachment (λ Mo-K_α, graphite monochromator, $\theta/2\theta$ scan). 2440 reflections with $F^2 > 1.9\sigma(F^2)$ were used for solution of the structure. Coordinates of the Re atoms were determined from the three-dimensional Patterson function, and all other non-hydrogen atoms were localized by successive syntheses of electron density. The structure was refined by the anisotropic full-matrix least-squares technique to $R = 0.052$.

The molecular geometry, main bond lengths and angles are shown in Fig. 1; atomic coordinates, bond lengths, bond angles and equations of some planar molecular fragments are given in Tables 3—6.

The Re(2) atom is coordinated with a η^5-Cp ligand and two carbonyls as well as with a monodentate vinylidene ligand, forming a metalallene system Re(2)=C(8)=C(7). The Re(1) atom is also coordinated with a η^5-Cp ligand and two car-

TABLE 3

ATOMIC COORDINATES (X 10^4)

Atom	x	y	z
Re(1)	2438.7(6)	6397.6(12)	727.6(5)
Re(2)	156.2(6)	9355 (1)	—1594.7(5)
C(1)	3596 (17)	6484 (37)	880 (15)
C(2)	2695 (12)	731 (23)	1655 (13)
O(1)	4297 (9)	6431 (21)	916 (9)
O(2)	2808 (9)	7839 (20)	2259 (9)
C(3)	—208 (13)	7293 (32)	—2037 (12)
C(4)	—182 (13)	8644 (29)	—748 (13)
O(3)	—388 (9)	6030 (20)	—2275 (9)
O(4)	—377 (9)	8202 (19)	—259 (8)
C(5)	3121 (12)	3810 (25)	4600 (11)
C(6)	2384 (12)	8552 (26)	—67 (11)
C(7)	2006 (13)	8050 (26)	—827 (11)
C(8)	1246 (14)	8591 (27)	—1136 (11)
C(9)	3161 (12)	9721 (24)	59 (12)
C(10)	3292 (13)	10392 (24)	—576 (13)
C(11)	3965 (14)	11465 (30)	—475 (15)
C(12)	4462 (13)	11893 (25)	226 (15)
C(13)	4290(14)	1123(29)	848(13)
C(14)	3632(12)	10112(29)	749(12)
C(15)	2407(14)	6907(25)	—1238(12)
C(16)	3247(13)	6567(28)	—969(12)
C(17)	3606(13)	5334(29)	—1321(12)
C(18)	3135(14)	4516(28)	—1932(14)
C(19)	2281(15)	4854(26)	—2228(12)
C(20)	1926(13)	6027(27)	—1876(12)
C(21)	1141(13)	5192(28)	455(15)
C(22)	1427(15)	5040(33)	—158(13)
C(23)	2139(16)	4042(26)	52(15)
C(24)	2307(15)	3627(30)	844(19)
C(25)	1679(16)	4398(31)	1097(15)
C(26)	—57(15)	10871(30)	—2652(12)
C(27)	625(14)	11599(25)	—2092(12)
C(28)	320(15)	12191(26)	—1474(14)
C(29)	4404(15)	3187(27)	3289(13)
C(30)	—792(14)	11031(31)	—2421(14)

TABLE 4

BOND LENGTHS d (Å)

Bond	d(Å)	Bond	d(Å)
Re(1)—C(1)	1.90(3)	C(9)—C(10)	1.40(3)
Re(1)—C(2)	1.85(2)	C(10)—C(11)	1.41(3)
Re(1)—C(5)	2.21(2)	C(11)—C(12)	1.40(4)
Re(1)—C(6)	2.31(2)	C(12)—C(13)	1.40(4)
Re(1)—C(21)	2.32(2)	C(13)—C(14)	1.41(3)
Re(1)—C(22)	2.31(3)	C(9)—C(14)	1.36(3)
Re(1)—C(23)	2.30(2)		
Re(I)—C(24)	2.30(2)	C(15)—C(16)	1.39(3)
Re(I)—C(25)	2.31(3)	C(16)—C(17)	1.44(3)
		C(17)—C(18)	1.38(3)
Re(2)—C(3)	1.91(3)	C(18)—C(19)	1.42(3)
Re(2)—C(4)	1.94(2)	C(19)—C(20)	1.40(3)
Re(2)—C(8)	1.90(2)	C(20)—C(15)	1.44(3)
Re(2)—C(26)	2.30(2)		
Re(2)—C(27)	2.31(2)	C(21)—C(22)	1.39(4)
Re(2)—C(28)	2.35(2)	C(22)—C(23)	1.42(4)
Re(2)—C(29)	2.36(2)	C(23)—C(24)	1.49(4)
Re(2)—C(30)	2.34(2)	C(24)—C(25)	1.43(4)
		C(21)—C(25)	1.45(4)
C(5)—C(6)	1.41(3)	C(26)—C(27)	1.45(4)
C(6)—C(7)	1.46(3)	C(27)—C(28)	1.49(4)
C(6)—C(9)	1.59(3)	C(28)—C(29)	1.52(4)
C(7)—C(8)	1.33(3)	C(29)—C(30)	1.44(4)
C(7)—C(15)	1.50(3)	C(26)—C(30)	1.44(4)
C(1)—O(1)	1.17(4)		
C(2)—O(2)	1.19(3)		
C(3)—O(3)	1.14(3)		
C(4)—O(4)	1.13(3)		

bonyls and in addition a π-ethylenic fragment bonded to the C(7) of a metalallene system occupies one coordination site.

The metalallene system is linear with the double bond Re=C(8) (1.90(2) Å) considerably shorter than the ordinary Re—C σ-bonds (2.25 in $CpRe(CH_3)_2C_5H_5CH_3$ [6], 2.22 Å in p-$ClC_6H_4Re(CO)_5$ [7] and 2.32 Å in $CpRe(CO)_2BrCH_3$ [8]) and the sum of the single-bonded covalent radii of Re and C(sp^3) (2.297 Å [9]). In contrast, the length of the Re=C(8) double bond is close to the sum of the double-bonded radii of Re (1.24 Å [10]) and C(sp^2) (0.67 Å). The metalallene system was previously found in (η^5-C_5H_5)$Mn(CO)_2$(C=CHPh) [5] where the length of the Mn=C bond is 1.68(2) Å. Both in this complex and in IV the metalallene system is almost coplanar with the C(15)—C(20) phenyl ring (torsional angle around the C(7)—C(15) bond is 15°).

The central C(5)—C(8) fragment of the organic ligand is nonplanar, the torsion-

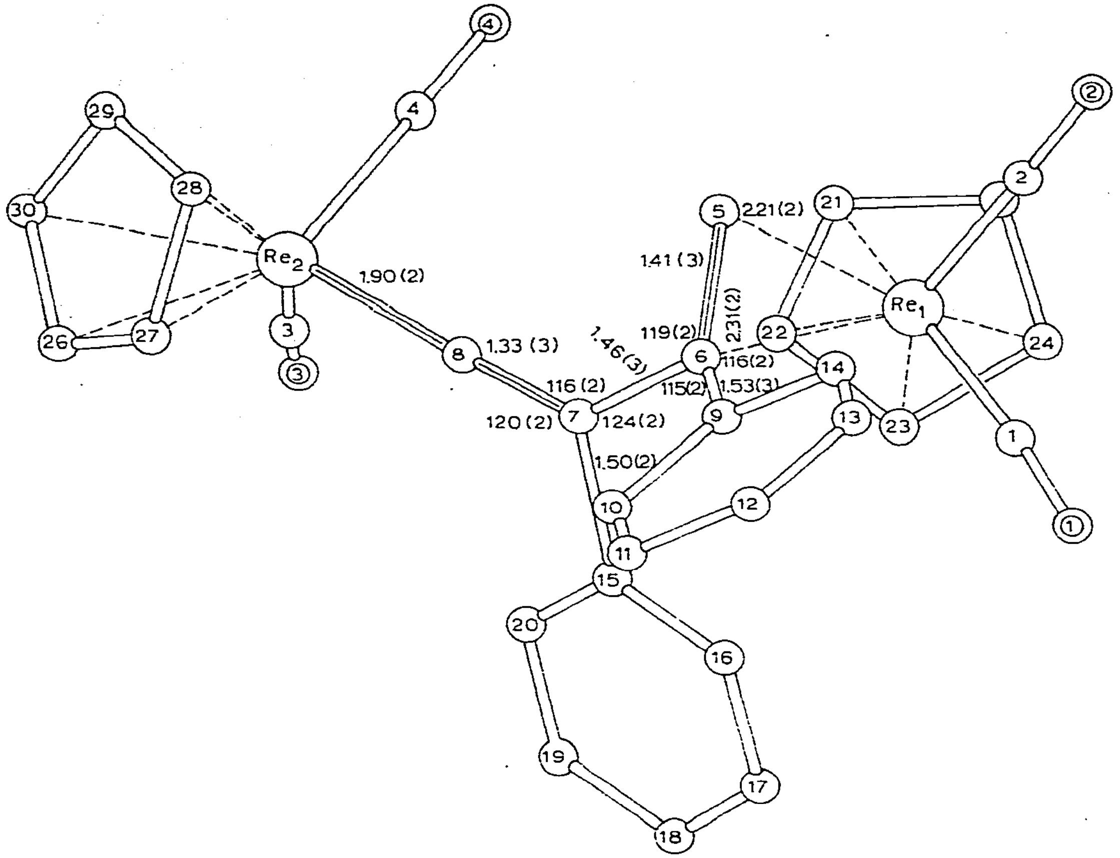

Fig. 1. The molecular structure of $Cp(CO)_2Re[C{=}C(Ph){-}C(Ph){=}CH_2]Re(CO)_2Cp$ with main bond lengths and angles.

al angle around the C(6)—C(7) bond being 29.7°, and the phenyl rings are turned relative to the C(6)—C(7)—C(8) and C(5)—C(6)—C(7) planes by 18° and 41° respectively; the dihedral angle between the phenyl rings is equal to 73°. The lengths of the C(7)—C(15) and C(6)—C(9) bonds are 1.50(3) and 1.59(3) Å respectively and presumably exclude conjugation of the phenyl rings with the butadiene C(5)—C(8) fragment.

The rest of the bond lengths and angles are normal, the average Re(2)—C(Cp), Re(1)—C(Cp) and C—C distances in the Cp ligands are 2.31(2), 2.33(2) and 1.46(4) Å respectively. Due to coordination with Re(1), the C(5)=C(6) distance is increased to 1.41(3) Å as compared with 1.33(3) Å for the non-coordinated C(8)=C(7) bond. The distances Re(1)—C(5) (2.21(2) Å and Re(1)—C(6) (2.31(2) Å) differ considerably. The bond lengths Re—CO vary from 1.85(2) to 1.94(2) Å and C—O bonds from 1.13(3) to 1.17(4) Å in length.

TABLE 5

BOND ANGLES, ω (degrees)

Angle	ω	Angle	ω
Re(1)—C(1)—O(1)	173(2)	C(15)—C(16)—C(17)	119(2)
Re(1)—C(2)—O(2)	175(2)	C(16)—C(17)—C(18)	121(2)
Re(1)—C(6)—C(7)	111(1)	C(17)—C(18)—C(19)	121(2)
C(1)—Re(1)—C(2)	84(1)	C(18)—C(19)—C(20)	118(2)
C(3)—Re(2)—C(4)	88(1)	C(19)—C(20)—C(15)	122(2)
C(5)—Re(1)—C(6)	36.4(7)	C(20)—C(15)—C(16)	119(2)
C(5)—C(6)—C(7)	119(2)		
C(5)—C(6)—C(9)	116(2)	C(21)—C(22)—C(23)	107(2)
C(6)—C(7)—C(8)	116(2)	C(22)—C(23)—C(24)	108(2)
C(6)—C(7)—C(15)	124(2)	C(23)—C(24)—C(25)	107(2)
C(6)—C(9)—C(10)	118(2)	C(24)—C(25)—C(20)	105(2)
C(6)—C(9)—C(14)	121(2)	C(25)—C(21)—C(22)	112(2)
C(7)—C(15)—C(16)	120(2)		
C(7)—C(15)—C(20)	121(2)	C(26)—C(27)—C(28)	109(2)
C(7)—C(6)—C(9)	115(2)	C(27)—C(28)—C(29)	105(2)
C(8)—C(7)—C(15)	120(2)	C(28)—C(29)—C(30)	108(2)
Re(2)—C(8)—C(7)	179(3)	C(29)—C(30)—C(26)	110(2)
Re(2)—C(3)—O(3)	176(2)	C(30)—C(26)—C(27)	108(2)
Re(2)—C(4)—O(4)	179(2)		
C(9)—C(10)—C(11)	117(2)		
C(10)—C(11)—C(12)	122(2)		
C(11)—C(12)—C(13)	119(2)		
C(12)—C(13)—C(14)	119(2)		
C(13)—C(14)—C(9)	123(2)		
C(14)—C(9)—C(10)	123(2)		

TABLE 6

EQUATIONS $Ax + By + Cz - D = 0$ OF PLANAR FRAGMENTS IN THE IV MOLECULE

Plane	*A*	*B*	*C*	*D*
C(21)—C(22)—C(23)—C(24)—C(25)	−0.5461	−0.8322	−0.0961	−4.638
C(26)—C(27)—C(28)—C(29)—C(30)	−0.2026	0.8824	−0.4247	9.9368
C(9)—C(10)—C(11)—C(12)—C(13)—C(14)	0.5899	−0.7757	−0.2243	−2.6775
C(15)—C(16)—C(17)—C(18)—C(19)—C(20)	0.1597	0.6937	−0.7023	7.0848

Experimental

Argon-saturated dry solvents were used. All operations were carried out under argon atmosphere. Photochemical reactions were run in a quartz apparatus equipped with a cooling jacket using a PK-7 mercury lamp (1000 W). Chromatography was performed on a column packed with silica (Chemapol, Czechoslovakia). IR spectra were measured with a UR-20 (Zeiss) instrument. Raman spectra were obtained on a PHO Coderg spectrometer with He/Ne-laser excitation. Mass spectra were obtained using a AEJ MS-30 instrument with a DS-50 data processing system at injection temperature 110°C, source temperature 200°C and ionization voltage 70 V. Resolution was 1000 for both beams.

^{13}C MNR spectra in CH_2Cl_2 were recorded with a Brucker HX-90 spectrometer (22.635 MHz).

Reaction of CpRe(CO)$_3$ with PhC≡CH

A mixture of 0.67 g (0.002 mol) of CpRe(CO)$_3$, 0.45 g (0.004 mol) of PhC≡CH and 40 ml of THF was UV irradiated at 5°C for 7 h with stirring. After evaporation of the solvent in vacuo a red-brown oil was obtained that was extracted with petroleum ether (b.p. 35—40°C). The extract was chromatographed on a silica column giving the following fractions: with petroleum ether colorless, (1); with ether/petroleum ether mixtures, (1 : 100) bright pink, (2), (1 : 50) yellow (3), (1 : 10) light red, (4), and (1 : 4) again yellow, (5).

Fraction (1) contained 0.5 g of a mixture of CpRe(CO)$_3$ and PhC≡CH. After evaporation of the solvent from fraction (2) the dry residue was recrystallized from petroleum ether to yield 0.12 g (12%) of CpRe(C=CHPh)(CO)$_2$ (II) as red plate-like crystals, m.p. 75—76°C. (Found: C 44.08; H, 2.47; Re, 44.73. $C_{15}H_{11}O_2Re$ calcd.: C, 43.99; H, 2.71; Re, 45.47%).

Removal of the solvent from fraction (3) and recrystallization of the solid residue from ether/petroleum ether mixture (1 : 25) rendered 0.04 g (3%) of Cp$_2$Re$_2$(μ-C=CHPh)(CO)$_4$ (III) as yellow crystals, m.p. 148—149°C. (Found: C, 37.35; H, 2.08; Re, 50.98. $C_{22}H_{16}O_4Re_2$ calcd.: C, 36.84; H, 2.25; Re, 51.96%).

Fraction (4) yielded, after evaporation of the solvent and recrystallization from ether, 0.09 g (4.3%) of Cp$_2$Re$_2$[C=C(Ph)C(Ph)=CH$_2$](CO)$_4$ (IV) as light red crystals, m.p. 150—152°C. (Found: C, 44.51; H, 2.96; Re, 45.13. $C_{30}H_{22}O_4Re_2$ calcd.: C, 43.99; H, 2.71; Re, 45.47%).

Fraction (5) rendered 0.04 g (3%) of Cp$_2$Re(CO)$_5$ (V) as yellow crystals identified by the IR spectrum of its solution in cyclohexane: ν(C≡O) 1994, 1958, 1925, 1905; ν(C=O) 1745 cm^{-3}. Ref. [11] in cyclohexane: ν(C≡O) 1992, 1956, 1923, 1904; ν(C=O) 1740 cm^{-1}.

Interaction of CpRe(C=CHPh)(CO)$_2$ (II) with a water/alcohol/base

To a pink solution of 0.1 g of II in 20 ml of MeOH/THF mixture (3 : 1) a mixture of 0.05 g of KOH, 10 ml of H$_2$O and 10 ml of MeOH was added dropwise. After stirring for 0.5 h at 65°C the reaction mixture turned yellow, the solvent was then evaporated and the dry residue extracted with petroleum. The extract was evaporated yielding yellow crystals of Cp$_2$Re$_2$(μ-C=CHPh)(CO)$_4$ (III) identified by IR spectroscopy and TLC.

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