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DIPHENYLBENCHROTRENYLPHOSPHINE – A NEW TWO-ELECTRON LIGAND IN CARBONYL-CONTAINING π-AROMATIC CHROMIUM AND MANGANESE COMPLEXES AND IN CARBONYLS OF GROUP VIB METALS

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Summary

Photochemical substitution of CO ligands in $C_6H_6Cr(CO)_3$, $C_5H_5Mn(CO)_3$ and $M(CO)_6$ (M = Cr, Mo, W) for $PPh_2C_6H_5Cr(CO)_3$ ligands was used to synthesize novel complexes: $C_6H_6Cr(CO)_2PPh_2C_6H_5Cr(CO)_3$, $C_5H_5Mn(CO)_2PPh_2C_6H_5Cr(CO)_3$ and $M(CO)_5PPh_2C_6H_5Cr(CO)_3$.

The complexes obtained have been characterized by elemental analysis, IR, ^1H, ^{13}C, ^{31}P NMR, and mass spectra.

The protonation reaction and the hydrogen isotopic exchange of arenechromium carbonyl complexes in acid media have been studied.

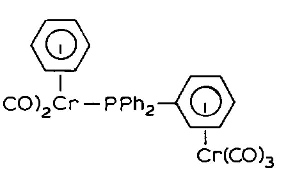
The ^{13}C NMR spectra of $M(CO)_5PPh_2C_6H_5Cr(CO)_3$ show that the nature of the central metal atom influences the chemical shifts of the carbon nuclei of the carbonyl groups, shielding these atoms, increasing in the series W $\leqslant$ Mo < Cr.

Introduction

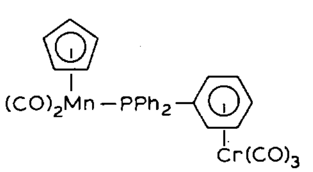
At present, a considerable number of transition-metal complexes with various phosphine ligands are known. However, knowledge of complexes with organometal phosphines is scant [1,2]. It was therefore of interest to use diphenylbenchrotrenylphosphine, $PPh_2C_6H_5Cr(CO)_3$, as a two-electron ligand and investigate its influence on the properties and reactivity of transition-metal complexes. This paper reports the results obtained.

Results and discussion

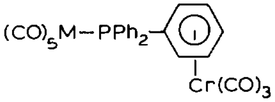
Derivatives of benchrotrene (Ia), cymantrene (II) and carbonyls of Group VIB metals (IIIa, b, c) were synthesized by photochemical substitution of the CO ligand in the starting complexes for $PPh_2C_6H_5Cr(CO)_3$.



(Ia)



(II)



(III)

M = Cr (a)
M = Mo (b)
M = W (c)

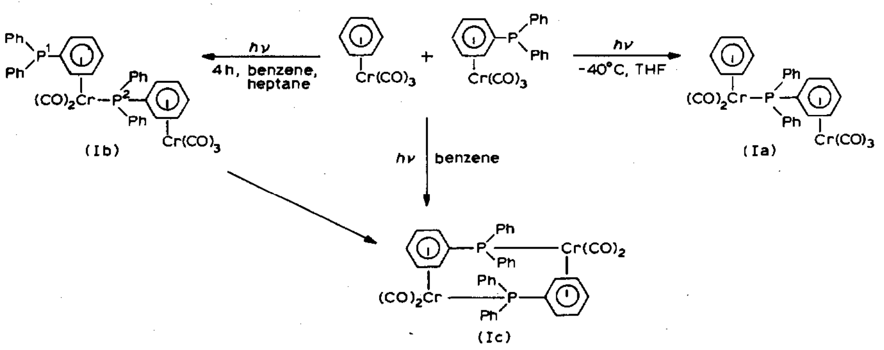
It was discovered that in the process the interaction of $PPh_2C_6H_5Cr(CO)_3$ with benchrotrene can take different directions depending on the reaction conditions, yielding complexes Ia, Ib and Ic (Scheme 1). Complexes Ia and Ib have never been obtained before; complex Ic has been obtained by boiling $Cr(CO)_6$ with Ph_3P in decalin [3].

Diphenylbenchrotrenylphosphine, $PPh_2C_6H_5Cr(CO)_3$, was obtained from the reaction of a lithium derivative of benchrotrene with PPh_2Cl in THF at −30°C (Scheme 2).

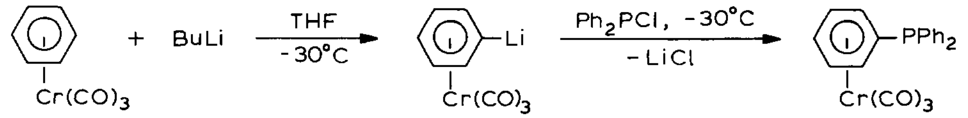
This method differs from the previously described one [4] in that there is no need for the additional stage of benchrotrene mercuration.

The complexes obtained are solid, crystalline substances, yellow in colour, rather stable in air, and readily soluble in most organic solvents. The compositions and structures of complexes I–III were obtained on the basis of their mass spectra, IR, ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra. Table 1 contains the relevant physicochemical constants of the complexes obtained and, for comparison, those of complexes with a PPh_3 ligand.

Four absorption bands were observed in the IR spectra of complexes Ia, II and III(a–c) in the region of CO-group stretching modes. For Ia, we assigned two bands (1840 and 1898 cm^{-1}) to the stretching modes of the $C_6H_6Cr(CO)_2$ moiety CO groups, and the other two (1925 and 1990 cm^{-1}) to the stretching modes of the phosphine ligand $Cr(CO)_3$ group. For complex II, absorption bands with $\nu(CO)$ 1876 and 1940 cm^{-1} were assigned to the $C_5H_5Mn(CO)_2$ moiety, and those with $\nu(CO)$ 1910 and 1981 cm^{-1} to the $Cr(CO)_3$ moiety. The data in Table 1 indicate that the substitution of one phenyl group in PPh_3 for $C_6H_5Cr(CO)_3$ leads to a 6–15 cm^{-1} shift in the stretching mode frequency of the $M(CO)_2$ moiety CO groups in complexes Ia and II into the short wave region. In the ^{1}H NMR spectra the



SCHEME 1



SCHEME 2

π-aromatic ligand proton signals in Ia and II are shifted downfield by 0.13–0.05 ppm, compared to the signals of the corresponding protons in complexes with a PPh_3 ligand. The IR and ^1H NMR data signify a somewhat lower electron-donating capacity of the $PPh_2C_6H_5Cr(CO)_3$ ligand with respect to PPh_3. A similar picture is true for the ^{13}C NMR spectra of these complexes (Table 2). The signals of the carbon nuclei in the π-aromatic ligands in Ia and II are shifted by 1.52 and 0.57 ppm, respectively, into the weak-field with respect to the positions of the analogous signals of complexes with a PPh_3 ligand.

π-Aromatic complexes of transition metals with phosphine ligands are known to be easily protonated in acid media at the metal atom [7–9]. In this respect it appeared interesting to investigate the behaviour of complexes Ia, II and Ib in acid media.

The IR spectrum of complex Ia (Table 3) in acid medium contained, along with the absorption bands ν(CO) of the non-protonated form (1840 and 1895 cm^{-1}), ν(CO) absorption bands shifted into the short wave region by 100–80 cm^{-1} (1940 and 1975 cm^{-1}). The size of this shift [8] indicates that complex Ia is protonated at the chromium atom, coordinated with the phosphine ligand. Complex II cannot be protonated under these conditions.

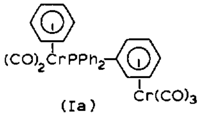
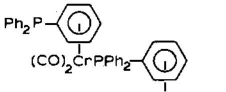
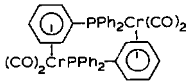
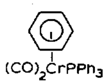
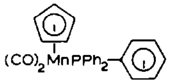
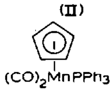
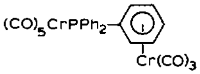
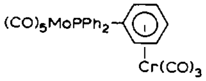
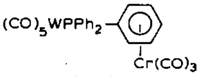
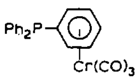
Protonation of complex Ia in acid medium is also confirmed by the ^{31}P NMR data (Table 4). In a neutral solvent, the ^{31}P NMR spectrum of Ia contained a singlet with δ 95.36 ppm. The ^{31}P $\{^1H\}$NMR spectrum in an acid medium contained two signals, δ 98.11 and δ 79.68 ppm. In the spectrum taken without proton decoupling, the signal at δ 79.68 ppm took the form of a doublet with spin coupling $J(^{31}P(Cr)–^1H)$ 78.12 Hz. The size of the shift of the phosphorus nucleus signal and the value of spin coupling [9] indicate protonation at the chromium atom. In the ^{31}P NMR spectrum of this complex, a singlet at δ 96.42 ppm was observed, which in acid medium was shifted downfield by approximately 4 ppm (solvent influence), while in the ^{31}P NMR spectrum, the signal remained unchanged. This confirms the conclusion drawn on the basis of the IR spectra that complex II cannot be protonated at the Mn atom under these conditions.

Two protonation sites are possible for complex Ib in an acid medium: at the uncoordinated phosphorus atom of the substituent in the π-arene ligand, and at the metal atom. In neutral medium, the ^{31}P NMR spectrum of Ib contained two signals: one of the uncoordinated phosphorus atom at δ -7.20 ppm, and one of the phosphorus atom coordinated with the chromium atom at δ 86.37 ppm. In acid medium, the ^{31}P NMR spectrum contained two groups of signals, pertaining to the protonated (δ 70.56 and 11.82 ppm) and the non-protonated (δ 78.74 and δ 8.77 ppm) phosphorus atoms. In the ^{31}P $^1\{H\}$NMR spectrum, only the signal at δ 11.82 ppm took the form of a doublet with spin coupling $J(^{31}P–^1H)$ 322.9 Hz, and the remaining signals were left unchanged. The value of spin coupling indicates that Ib is protonated at the phosphorus atom with the free electron pair.

(Continued on p. 192)

TABLE 1

SOME PHYSICOCHEMICAL PROPERTIES OF COMPLEXES Ia–c, II AND IIIa–c

Complex	ν(CO) (cm^{-1}) (CH$_2$Cl$_2$)	π-Aromatic ligand	π-Benzene ring in phosphine ligand δ, ppm (C$_6$D$_6$)
 (Ia)	1840, 1898, 1925, 1990	4.23	5.47(*o*), 5.59(*p*), 4.72(*m*)
 (Ib)	1855, 1900w, 1978	4.76(o), 4.56(p) 4.43(m)	5.46 (multiplet centre)
 (Ic)	1850, 1900	–	5.63–4.77
	1825, 1884	4.10	–
 (II)	1876, 1940, 1910, 1981	4.19 [a]	5.44(*o*), 4.92(*m* + *p*) [a]
	1870, 1934	4.14 [a]	–
 (IIIa)	1920, 1952, 1988, 2072	–	5.46(*o*), 5.54(*p*) [a], 4.99(*m*) [a]
(CO)$_5$Cr PPh$_3$ [5]	1944, 1989, 2070	–	–
 (IIIb)	1910, 1955, 1980, 2080	–	5.18(*o*), 5.43(*p*), 5.05(*m*) [a]
(CO)$_5$MoPPh$_3$ [5]	1951, 1990, 2078	–	–
 (IIIc)	1920, 1950, 1987, 2080	–	5.44(*o*), 5.52(*p*) [a], 5.00(*m*) [a]
(CO)$_5$WPPh$_3$ [5]	1943, 1981, 2074	–	–
	1911, 1978	–	4.77(*o*), 4.45(*p*), 4.18(*m*) 5.34(*o*), 5.16 (*m* + *p*) [a]

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[a] ^{1}H NMR spectra taken in CS$_2$.

TABLE 2

^{13}C NMR DATA OF COMPLEXES Ia AND II

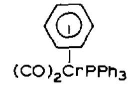
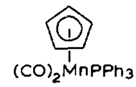
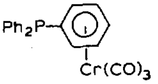
Complex	δ, ppm (CH_2Cl_2)									
	π-Aromatic ligand	Phosphine ligand								CO
		π-Benzene ring				Ph				
		C(1)	C(2,6)	C(3,5)	C(4)	C(1)	C(2,6)	C(3,5)	C(4)	
(Ia) 	90.65 89.13			87.82–99.60 –		168.00 139.26	129.99 128.13	129.03 127.13	133.34 132.25	221.00 200.87
(II) 	82.97 82.40		96.31	87.91 –	99.56	138.21	130.26	128.47 –	130.29	231.53 233.30 [6]
	–	104.66	95.50	92.39	99.72	137.10	130.70	130.00	135.11	233.61

TABLE 3

IR SPECTRAL DATA OF COMPLEXES Ia AND II IN NEUTRAL AND ACID MEDIA

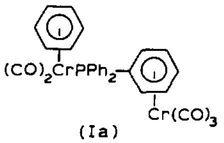
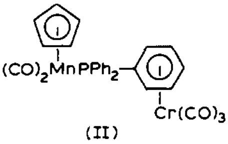
Complex	Medium	ν(CO) (cm^{-1})	
		$M(CO)_2$	$M(CO)_3$
 (Ia)	CH_2Cl_2 CH_2Cl_2/CF_3COOH 1/1	1840, 1895 1840, 1895, 1940, 1975	1925, 1990 1940, 2010
 (II)	CH_2Cl_2 CH_2Cl_2/CF_3COOH 1/1	1878, 1940 1885, 1950	1918, 1980 1950, 1995

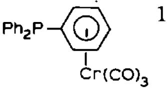
TABLE 4

^{31}P NMR SPECTRAL DATA OF COMPLEXES Ia, II AND Ib IN NEUTRAL AND ACID MEDIA

Complex	δ(^{31}P) ppm			J(^{31}P Cr–^{1}H) (Hz)	J(^{31}P–H) (Hz)
	CH_2Cl_2	CH_2Cl_2/CF_3COOH 1/1			
	^{31}P–{^{1}H}	^{31}P–{^{1}H}	^{31}P		
Ia	95.36	79.68 98.11	80.75, 78.66 99.45	78.12	–
II	96.42	100.32	100.32	–	–
Ib	86.37 –7.20	78.74, 70.56 8.77, 11.82	78.75, 70.56 8.85, 14.84, 10.84	–	322.90

TABLE 5

^{13}C NMR DATA OF COMPLEXES IIIa–c AND $(CO)_5MPPh_3$ (M = Cr, Mo, W)

Complex	δ, ppm (CH_2Cl_2)									
	π-Benzene ring in phosphine ligand				Ph				CO in phosphine ligand	CO in $M(CO)_5$
	C(1)	C(2,6)	C(3,5)	C(4)	C(1)	C(2,6)	C(3,5)	C(4)		
IIIa	98.22	94.21	86.17	97.22	131.56	128.99	127.08	130.45	229.08	214.28
$(CO)_5CrPPh_3$										218.4 [10]
IIIb	100.05	95.57	89.57	97.22	136.50	134.18	131.84	135.38	231.12	205.47
$(CO)_5MoPPh_3$										205.8 [11]
IIIc	100.70	96.17	88.35	99.34	135.80	131.16	129.01	133.22	231.45	197.22
$(CO)_5WPPh_3$										198.0 [12]
	104.66	95.50	92.39	99.72	137.10	130.70	130.00	135.11	233.61	–

The effect of $PPh_2C_6H_5Cr(CO)_3$ on the reactivity of the π-aromatic ligand in the complexes under examination was studied in the hydrogen isotopic exchange (HIE) reaction of Ia in CF_3COOD. The ^1H NMR spectra were used to show that only the protons of the π-benzene ring in Ia participate in the HIE reaction. The rate of the HIE reaction of Ia (k 1.0×10^{-4} s^{-1}) was 2.5 times slower than that of $C_6H_6Cr(CO)_2PPh_3$ (k 2.5×10^{-4} s^{-1}). This confirms the conclusion backed by spectral data that the substitution of a Ph group for $C_6H_5Cr(CO)_3$ reduces the electron-donating capacity of the phosphine ligand.

On the basis of the ^{13}C NMR data of complexes IIIa–c, it is possible to determine the changes that take place in the $PPh_2C_6H_5Cr(CO)_3$ phosphine ligand upon its coordination with the $M(CO)_5$ group (M = Cr, Mo, W). Comparison of all the chemical shifts of the ^{13}C carbon nuclei in the ligand π-benzene ring of complexes IIIa–c and the uncoordinated $PPh_2C_6H_5Cr(CO)_3$ shows (Table 5) that the greatest change in the value of the chemical shift in the process of coordination occurs for the *meta*-carbons of the C_6H_5Cr moiety. The signals of the ^{13}C carbon atoms in the *meta* position are shifted upfield by 3–6 ppm, while the signals of the ^{13}C carbon nuclei in the *ortho* and *para* positions have average shifts of 0.5–3 ppm. Greater screening of carbon *meta*-atoms, in comparision with the *ortho* and *para* atoms, in π-arene ligands with electron-accepting substituents is known [13]. Moreover, the nature of the central metal atom in the $M(CO)_5$ moiety influences the position of the signal of the $Cr(CO)_3$ moiety carbon nuclei, whose shielding increases in the series Cr > Mo $\geqslant$ W.

Experimental

All operations regarding the synthesis of complexes were conducted under argon. Solvents were dried and distilled in argon over $LiAlH_4$ prior to use. IR spectra were recorded on a UR-20 spectrophotometer, ^{13}C NMR and ^1H NMR spectra on a Bruker WP200-SY spectrometer (200 MHz), and ^{31}P NMR spectra on a Bruker-Physik HX-90 spectrometer (36, 43 MHz). A quartz 375 W bulb was used for UV irradiation.

$PPh_2C_6H_5Cr(CO)_3$

0.5 g (2.2 mmol) of $C_6H_6Cr(CO)_3$ was placed in a double-neck flask under vacuum and filled with argon. 30 ml of THF was then distilled into the reaction flask. 20 ml of 0.7 *N* BuLi solution was added dropwise, while stirring energetically until the solution cooled to −40°C. After 40 min, 2.8 g (13 mmol) of PPh_2Cl was added dropwise. The mixture was stirred for another hour at −40°C, and then the temperature of the reaction mixture was gradually brought up to 20°C. The solvent was distilled off in vacuo, and the residue subjected to chromatography on Al_2O_3. 0.56 g (60%) of oily yellow crystals of $PPh_2C_6H_5Cr(CO)_3$ was obtained. Mass spectrum, *m/e*, (I): 398(4.2) M^+; 314(82.6) M-3CO$^+$; 262(69.5) $PPh_2C_6H_5{}^+$; 52(4.5)Cr^+. ^{1}H NMR spectrum (δ, ppm): 4.17d(2H *ortho*), 4.45t(H *para*), 4.18t(2H *meta*), 7.42–6.96m(10H–Ph).

$C_6H_6Cr(CO)_2PPh_2C_6H_5Cr(CO)_3$ (Ia)

0.10 g (0.5 mmol) of $C_6H_6Cr(CO)_3$ in 40 ml of THF was irradiated with UV light at −40°C for 3 h. To the resultant dark-brown solution, 0.20 g (0.5 mmol) of $PPh_2C_6H_5Cr(CO)_3$ was added in 10 ml of THF while stirring. The reaction mixture was stirred for another hour at −40°C, then brought to room temperature; the colour of the solution changed from dark brown to orange. The solvent was distilled off in vacuo, and the solid residue was subjected to chromatography on Al_2O_3. 0.10 g (37%) of Ia was obtained, yellow crystals, m.p. (decomp.) 170°C. Elemental analysis: Found: C, 59.01; H, 3.49; Cr, 17.54; P, 5.25, $C_{29}H_{21}Cr_2PO_5$ calcd.: C, 59.59; H, 3.59; Cr, 17.81; P, 5.31%. ^{1}H NMR spectrum (δ, ppm, C_6D_6): 4.23d(6H arene), 5.47d(2H *ortho*); 4.72t(2H *meta*); 5.59t(1H *para*); 7.08–7.72m(10H–Ph).

$PPh_2C_6H_5Cr(CO)_2PPh_2C_6H_5Cr(CO)_3$ (Ib)

0.10 g (0.5 mmol) of $C_6H_6Cr(CO)_3$ and 0.20 g (0.5 mmol) of $PPh_2C_6H_5Cr(CO)_3$ in a benzene/heptane mixture 1/2 were subjected to UV irradiation at room temperature for 4 h. After termination of the reaction, the solvent was distilled in vacuo and the residue was subjected to chromatography on Al_2O_3. 0.21 g (58%) of Ib was obtained, as yellow crystals, m.p. (decomp.) 200°C. Mass spectrum, *m/e* (I): 628(1,4)M-5CO$^+$; 314(17) $PPh_2C_6H_5Cr^+$; 262(29)$PPh_2C_6H_5{}^+$; 52(100)Cr^+. ^{1}H NMR spectrum (δ, ppm, C_6D_6): 4.43t(2H *meta*), 4.56t(1H *para*), 4.76d(2H *ortho*), 5.46m(5H); 6.88–8.01m(2OH, Ph).

$[PPh_2C_6H_5Cr(CO)_2]_2$ (Ic)

0.30 g (0.8 mmol) of $PPh_2C_6H_5Cr(CO)_3$ in 30 ml of benzene was subjected to UV irradiation at room temperature for 20 h. After termination of the reaction, the solvent was distilled off in vacuo and the solid residue was subjected to chromatography on Al_2O_3. 0.36 g (60%) of yellow crystals (Ic), m.p. (decomp.) 87°C were obtained. Analysis: C, 64.80; H, 4.17; P, 8.33, $C_{40}H_{30}P_2Cr_2O_4$ calcd.: C, 64.86; H, 4.05; P, 8.38%.

$C_5H_5Mn(CO)_2PPh_2C_6H_5Cr(CO)_3$ (II)

0.30 g (1.4 mmol) of $C_5H_5Mn(CO)_3$ in 30 ml of THF was irradiated with UV light for 6 h at 5°C. To the resultant red-violet solution, 0.41 g (1.0 mmol) of $PPh_2C_6H_5Cr(CO)_3$ was added in 10 ml of THF while stirring. The reaction mixture was stirred at 5°C for another hour and then left overnight at 20°C. After chromatography on Al_2O_3, 0.28 g (58%) of orange crystals (II) were obtained, m.p.(decomp.) 200°C. Mass spectrum, m/e (I): 574(2,5) M^+; ions corresponding to consecutive CO group split off; 382(100) $C_5H_5MnPPh_3{}^+$; 316(20,5)Ph_3PCr^+. ^1H NMR spectrum(δ, ppm, CS_2): 4.19d(5H arene); 5.44d(2H *ortho*); 4.92m(3H *meta para*); 7.75–7.01m(1OH Ph).

$(CO)_5CrPPh_2C_6H_5Cr(CO)_3$ (IIIa)

0.25 g (1.1 mmol) of $Cr(CO)_6$ in 40 ml of THF was irradiated with UV light for 4.5 h at 5°C. To the resultant orange solution, 0.3 g (0.8 mmol) of $PPh_2C_6H_5Cr(CO)_3$ was added in 10 ml of THF while stirring. The reaction mixture was stirred at 5°C for another hour and then left overnight at 20°C. The solvent was distilled off and the solid residue subjected to chromatography on Al_2O_3. Yield: 0.2 g (46%) of light yellow crystals (IIIa), m.p.(decomp.) 142–144°C. Mass spectrum, m/e (I): 590(31)M^+; 506(4)M–$3CO^+$; 366(16)M–$8CO^+$; 314(73)$CrPPh_3^+$; 52(100)Cr^+. ^1H NMR spectrum (δ, ppm, CS_2): 5.46d(2H *ortho*); 5.54t(1H *para*); 4.99t(2H *meta*); 7.59–7.18m(10H–Ph).

$(CO)_5MoPPh_2C_6H_5Cr(CO)_3$ (IIIb)

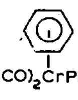
IIIb was obtained similarly to IIIa from 0.3 g (1.1 mmol) of $Mo(CO)_6$ and 0.4 g (1.0 mmol) of $PPh_2C_6H_5Cr(CO)_3$. Yield: 0.4 g (45%) of light orange, oily crystals (IIIb), m.p.(decomp.) 96°C. Mass spectrum, m/e (I): 494(3)M–$5CO^+$; 410(3)M–$8CO^+$; 384(37)Ph_3PMo^+; 314(97)Ph_3PCr^+. ^1H-NMR spectrum (δ, ppm, CS_2): 5.18d(2H *ortho*); 5.43t(1H *para*); 5.05t(2H *meta*); 7.22–7.41m(10H–Ph).

$(CO)_5WPPh_2C_6H_5Cr(CO)_3$ (IIIc)

IIIc was obtained similarly to IIIa from 0.34 g (0.9 mmol) of $W(CO)_6$ and 0.35 g (0.9 mmol) of $PPh_2C_6H_5Cr(CO)_3$. Yield: 0.23 g (38%) of greenish-yellow crystals (IIIc), m.p.(decomp.) 169–172°C. Mass spectrum, m/e (I): 638(27)M–$3CO^+$ products of consecutive CO group split off; 346(15)$WPPh_3{}^+$; 314(88)$CrPPh_3{}^+$; 184(100)W^+. ^1H NMR spectrum (δ, ppm, CS_2): 5.44d(2H *ortho*); 5.52t(1H *para*); 5.00t(2H *meta*); 7.22–7.41m(10H–Ph).

TABLE 6

DATA ON HYDROGEN ISOTOPIC EXCHANGE (equilibrium deuterium content = 82.0%.).

Complex	Time (h)	Deuterium content (atom.%)	k (HIE)×10^4 (s^{-1})
Ia	1.0	26.7	1.09
	2.0	42.0	1.00
	3.00	52.0	0.93
			k_{av} = 1.01
	1.5	66.0	2.98
	2.0	75.0	2.18
	2.5	81.0	2.28
			k_{av} = 2.48

Hydrogen isotopic exchange (HIE) in complex Ia

The HIE reaction of complex Ia and, for comparison, of $C_6H_6Cr(CO)_2PPh_3$, was conducted according to [1] at molar ratios of complex/CF_3COOD/C_6H_6 = 1/200/100. The deuterium content was determined, using ^1H NMR spectra, from the changes in the signal integral intensity of the π-benzene ring protons with respect to the signal integral intensity of the phenyl group protons in the phosphine ligand, which under the specified conditions cannot be substituted. The data on the deuterium exchange are given in Table 6.

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