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MASS SPECTROMETRY OF π-COMPLEXES OF TRANSITION METALS
I. THE INFLUENCE OF σ-BONDED LIGANDS ON THE FRAGMENTATION OF π-ALLYL DERIVATIVES OF IRON CARBONYL

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SUMMARY

Mass spectra of the π-allyliron complexes $RC_3H_4Fe(CO)_3X$ where X is a σ-ligand such as halogen or NO_3, have been studied. The fragmentation pattern indicates that decomposition proceeds principally through the rupture of Fe–CO and Fe–X bonds. The ratio of the intensities of the peaks characterising the ions formed from Fe–CO and Fe–X bond rupture depends on the nature of the substituent R and ligand X.

INTRODUCTION

Recently, the scope of organometallic compounds studied mass-spectrometrically has been extended considerably to include, inter alia, a number of π-allyl complexes of metals, *e.g.*, the homoligand complexes[1] $(C_3H_5)_nM$ where M is Ni, Pd, or Pt ($n=2$), Cr ($n=3$), Zr ($n=4$), the allylmetal halides (RC_3H_4PdX)[2,3] and $(RC_3H_4)_4Rh_2Cl_2$[2], and the complexes $C_3H_5Mo(CO)_2C_5H_5$[4] and $C_3H_5M(CO)_nC_5H_5$ where M is Ru, Fe ($n=1$), Mo, W ($n=2$)[5] containing an allyl and cyclopentadienyl ligand.

We have applied an electron impact technique to the study of the compounds $RC_3H_4Fe(CO)_3X$ (where X is a σ-bonded ligand), a compound of this type, *i.e.* $C_3H_5Fe(CO)_3I$, having been studied earlier by King[5]. Thus, compounds similar to the allylmetal carbonyl complexes, which are the last compounds listed above, have been subjected to the electron impact method. This method allows not only the principal fragmentation paths to be obtained but also enables the component processes to be visualised as a function of the group X, and of the substituent attached to the allyl ligand.

RESULTS AND DISCUSSION

The π-allyl complexes $RC_3H_4Fe(CO)_3X$ [(I), X=NO_3; (II), X=Cl; (III), X=Br; (IV), X=I; (a), R=H; (b), R=1-Me; (c), R=2-Me; (d), R=1-Ph; (e), R=2-Ph; (f), R=2-Br) decompose when subjected to electron bombardment. The molecular peaks (M^+) of the compounds (I) to (III) are either insignificant or are totally absent. For the iodides (IV), however, the M^+ peaks are quite intense (53% I_{max} for R=H,

TABLE I

THE m/e VALUES FOR THE MAJOR IONS IN THE MASS SPECTRA OF π-ALLYLIRON CARBONYL DERIVATIVES[a]

X	R		M+	φ_1	φ_2	φ_3	φ_4	φ_5	φ_6	φ_7
NO_3	H	(Ia)	243	181	112	174	153	215	84	146
			–	(16)	(7)	–	(60)	(3)	(19)	(2)
	1-Me	(Ib)	257	195	112	174	167	229	84	146
			–	(17)	(9)	(3)	(76)	(1)	(4)	–
	2-Me	(Ic)	257	195	112	174	167	229	84	146
			–	(10)	(10)	(1)	(37)	–	(9)	(1)
Cl	H	(IIa)	216, 218	181	112	147, 149	153	188, 190	84	119, 121
			(<0.01)	(21)	(4)	(2)	(81)	(28)	(5)	(6)
	1-Me	(IIb)	230, 232	195	112	147, 149	167	202, 204	84	119, 121
			(<0.01)	(18)	(19)	(2)	(69)	(11)	(25)	(2)
	2-Me	(IIc)	230, 232	195	112	147, 149	167	202, 204	84	119, 121
			(<0.01)	(12)	(10)	(3)	(42)	(5)	(20)	(8)
	1-Ph	(IId)	292, 294	257	112	147, 149	229	264, 266	84	119, 121
			(<0.1)	(3)	–	(4)	(19)	(4)	(3)	(10)
Br	H	(IIIa)	260, 262	181	112	191, 193	153	232, 234	84	163, 165
			(<0.1)	(16)	(5)	(4)	(54)	(33)	(12)	(10)
	1-Me	(IIIb)	274, 276	195	112	191, 193	167	246, 248	84	163, 165
			(0.7)	(17)	(13)	(1)	(75)	(24)	(26)	(16)
	2-Me	(IIIc)	274, 276	195	112	191, 193	167	246, 248	84	163, 165
			(0.2)	(12)	(12)	(1)	(48)	(6)	(14)	(15)
	1-Ph	(IIId)	336, 338	257	112	191, 193	229	308, 310	84	163, 165
			(<0.01)	(2)	(3)	–	(11)	(2)	(1)	(4)
	2-Ph	(IIIe)	236, 238	257	112	191, 193	229	308, 310	84	163, 165
			(<0.1)	(4)	(8)	–	(19)	(1)	(3)	(3)
	2-Br	(IIIf)	338, 340 342	259, 261	112	191, 193	231, 233	310, 312 314	84	163, 165
			–	(11)	(8)	(5)	(35)	(11)	(19)	(22)
	H	(IVa)	308	181	112	239	153	280	84	211
			(53)	(8)	(3)	(5)	(25)	(57)	(9)	(18)
	2-Me	(IVc)	322	195	112	239	167	294	84	211
			(29)	(11)	(8)	(8)	(43)	(51)	(3)	(4)

[a] The values in parentheses are relative intensities (% I_{max}).

20% I_{max} for R = 2-Me). Consequently, either iodine stabilises the molecular ion to a greater extent than Cl, Br, or NO_3 and/or the Fe–X bond is stronger when X is iodine. The molecular ions of (I) to (IV) decompose along two principal routes involving Fe–X and Fe–CO bond rupture and resulting in the φ_1 and φ_5 ions, respectively, (Scheme 1). Subsequently, the ions φ_1 successively lose three CO groups to form $[RC_3H_4Fe(CO)_n]^+$ ions where n is 2 to 0. The φ_5 ions on the other hand, successively lose two CO groups to form $[RC_3H_4Fe(CO)_nX]^+$ ions where n is 1 to 0. The mass spectra of (I) to (IV) in addition show the presence of $[Fe(CO)_n]^+$ and $[Fe(CO)_nX]^+$ ions (where n is 2 to 0), these ions being formed through the elimination of RC_3H_4 from $[RC_3H_4Fe(CO)_n]^+$ and $[RC_3H_4Fe(CO)_nX]^+$, respectively.

In addition to $[RC_3H_4Fe]^+$ (φ_{12}) peaks, all the compounds revealed intense (up to 90% I_{max}) peaks of the ions $[RC_3H_3Fe]^+$ (φ_{13}) which may result from φ_{12} by the loss of H and from φ_{14} by the loss of HX. When R is 1-CH_3, the ions probably correspond to butadiene- or cyclobutene-iron, and when R = 2-CH_3 to (trimethylene-

φ_8	φ_9	φ_{10}	φ_{11}	φ_{12}	φ_{13}	φ_{14}	φ_{15}	φ_{16}	φ_{17}	φ_{18}	φ_{19}
125	187	56	118	97	96	159	41	71	95	40	39
(37)	(15)	(43)	(3)	(100)	(15)	(38)	(53)	(33)	(61)	(21)	(78)
139	201	56	118	111	110	173	55	71	95	40	39
(51)	(11)	(50)	–	(100)	(7)	(32)	(69)	(9)	(5)	(8)	(30)
139	201	56	118	111	110	173	55	71	95	40	39
(24)	–	(35)	(1)	(100)	(8)	(10)	(79)	(27)	(49)	(15)	(4)
125	160, 162	56	91, 93	97	96	132, 134	41	71	95	40	39
(47)	(99)	(13)	(14)	(100)	(54)	(100)	(21)	(20)	(42)	(6)	(19)
139	174, 176	56	91, 93	111	110	146, 148	55	71	95	40	39
(45)	(20)	(48)	(6)	(100)	(51)	(22)	(47)	(4)	(8)	(12)	(15)
139	174, 176	56	91, 93	111	110	146, 148	55	71	95	40	39
(29)	(24)	(63)	(28)	(100)	(19)	(40)	(16)	(55)	(19)	(6)	(20)
201	236, 238	56	91, 93	173	172	208, 210	117	71	95	40	39
(31)	(16)	(7)	(20)	(64)	(36)	(43)	(100)	(5)	(4)	(9)	(2)
125	204, 206	56	135, 137	97	96	176, 178	41	71	95	40	39
(30)	(100)	(26)	(40)	(64)	(40)	(80)	(46)	(17)	(46)	(9)	(42)
139	218, 220	56	135, 137	111	110	190, 192	55	71	95	40	39
(41)	(90)	(100)	(54)	(100)	(97)	(70)	(82)	(11)	(12)	(24)	(47)
139	218, 220	56	135, 137	111	110	190, 192	55	71	95	40	39
(30)	(26)	(62)	(20)	(100)	(40)	(20)	(76)	(42)	(20)	(16)	(44)
201	280, 282	56	135, 137	173	172	252, 254	117	71	95	40	39
(18)	(8)	(8)	(4)	(34)	(17)	(10)	(100)	(4)	(4)	(2)	(5)
201	280, 282	56	135, 137	173	172	252, 254	117	71	95	40	39
(30)	(2)	(25)	(4)	(53)	(20)	(14)	(88)	(5)	(11)	(4)	(10)
203, 205	282, 284 286	56	135, 137	175, 177	174, 176	254, 256 258	119, 121	71	95	40	39
(22)	(32)	(36)	(100)	(65)	(6)	(34)	(5)	(3)	(10)	(60)	(60)
125	252	56	183	97	96	224	41	71	95	40	39
(16)	(98)	(30)	(100)	(50)	(60)	(75)	(20)	(13)	(27)	(4)	(20)
139	266	56	183	111	110	238	55	71	95	40	39
(39)	(97)	(14)	(24)	(100)	(49)	(98)	(18)	(28)	(11)	(5)	(10)

methane)-iron. Such processes are not observed for $[RC_3H_4Fe(CO)_n]^+$ and $[RC_3H_4Fe(CO)_nX]^+$, despite the fact that the (trimethylenemethane)-iron tricarbonyl system which could arise from these ions[6] is rather stable (the molecular ion $[C_4H_6Fe(CO)_3]^+$ was observed in the mass spectrum of this compound[7]).

Peaks corresponding to the $[CH_3Fe]^+$ (φ_{16}) ions are present in all the spectra such ions resulting from the one-step transformation of $[RC_3H_4Fe]^+$ (φ_{12}). This is confirmed by the presence of the corresponding metastable peaks in the spectra. The formation of φ_{16} may be due to the migration of one of the hydrogen atoms to the CH_2 group followed by C–C bond rupture and the elimination of RC≡CH. In addition, complexes containing the CH_3 group attached to the allyl may produce $[CH_3Fe]^+$ ions through the methyl migration to the iron atom, since compounds with $R = C_6H_5$ also display $[C_6H_5Fe]^+$ peaks.

King[3] has found that the intensity of φ_{16} is much higher than that reported in this study. His experiments were undertaken at temperatures at or above 200°, under

SCHEME I

The asterisks denote paths verified through the observation of peaks of the corresponding metastable ions.

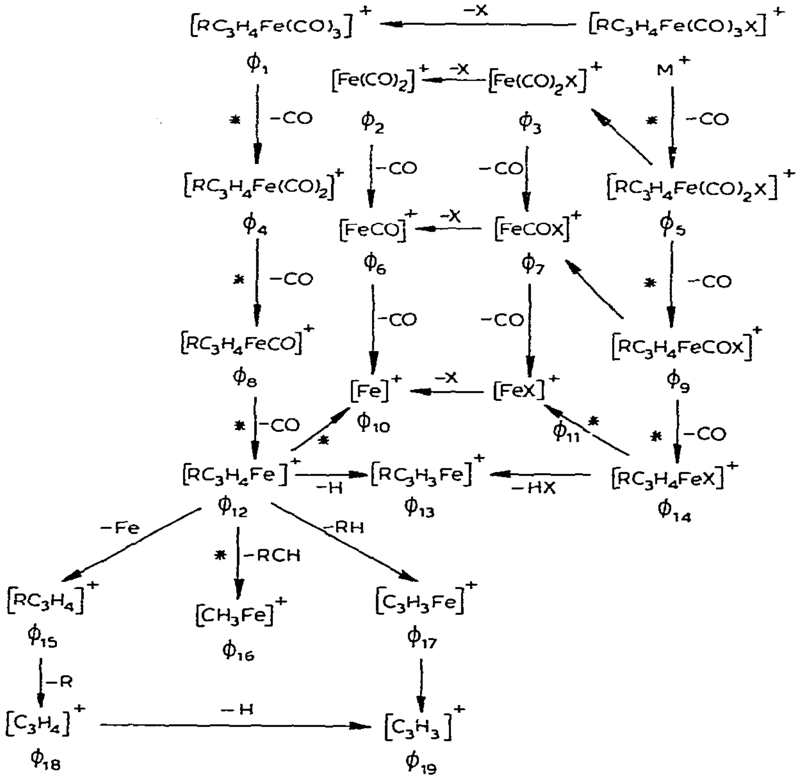

TABLE 2

THE φ_i/φ_j RATIOS IN THE MASS SPECTRA OF COMPOUNDS (I)–(IV)

φ_i/φ_j	R=H and X=				R=1-Me and X=				R=2-Me and X=			
	NO_3	Cl	Br	I	NO_3	Cl	Br	I	NO_3	Cl	Br	I
φ_1/φ_{11}	4.9	1.2	0.85	0.42	6.9	4.6	1.4	–	21.0	3.9	4.0	0.8
φ_1/φ_5	5.3	0.75	0.49	0.14	17.0	1.6	0.7	–	–	2.4	2.0	0.2
φ_4/φ_5	17.0	2.9	1.6	0.23	76.0	6.3	3.1	–	–	8.6	8.0	0.8
φ_8/φ_9	2.5	0.48	0.30	0.16	4.6	2.2	0.4	–	–	1.2	1.1	0.3
$\varphi_{12}/\varphi_{14}$	2.6	1.0	0.80	0.67	3.1	4.5	1.4	–	10	2.5	5.0	1.0
φ_9/φ_5	5.0	3.5	3.0	1.7	11.0	1.8	3.7	–	–	4.8	4.3	2.0
φ_{14}/φ_9	2.5	1.0	0.8	0.7	2.9	1.1	0.8	–	–	1.6	0.8	1.0
φ_4/φ_1	3.8	3.8	3.4	3.2	4.1	3.8	4.4	–	3.7	3.5	4.0	3.9
φ_8/φ_4	0.61	0.58	0.56	0.66	0.66	0.65	0.55	–	0.65	0.62	0.62	0.67
φ_{12}/φ_8	2.7	2.1	2.1	3.1	2.0	2.2	2.4	–	4.1	3.4	3.3	2.2
$\varphi_{16}/\varphi_{12}$	0.31	0.21	0.26	0.26	0.09	0.05	0.11	–	0.27	0.50	0.42	0.28
$\varphi_{11}/\varphi_{14}$	0.08	0.14	0.50	1.3	0	0.27	0.77	–	0.1	0.7	1.0	0.3

which conditions he also observed a greater number of fragments corresponding to cleavage of the organic moiety, *e.g.*, $[C_2H_2Fe(CO)_n]^+$ (n is 0 to 3), diallyl, and products of its decomposition. This may be due to thermolysis since the substrates are known to readily decompose at temperatures about 100°[8].

The overall fragmentation pattern is independent of the nature of X and R, although these affect the ratio of intensities significantly (Table 1). The ratio of the gross intensity of the peaks formed via the first path, *i.e.* via the elimination of X ($\varphi_1+\varphi_4+\varphi_8+\varphi_{12}+\varphi_{10}+\varphi_{16}=\varphi_{\mathrm{I}}$), to the gross intensity of the peaks formed via the second path, *i.e.* via the elimination of CO ($\varphi_5+\varphi_9+\varphi_{11}+\varphi_{14}=\varphi_{\mathrm{II}}$), increases over the series $\mathrm{I}<\mathrm{Br}<\mathrm{Cl}<\mathrm{NO_3}$, *i.e.*, with a decrease in the electronegativity of X (Table 2). A similar effect is found in the variation in the values $\varphi_{\mathrm{I}}/\varphi_5$, φ_4/φ_5, φ_8/φ_9, $\varphi_{12}/\varphi_{14}$, φ_9/φ_5, φ_{14}/φ_9. Consequently, the substituent X strongly affects the Fe–X bond strength and the stability of the ion $[RC_3H_4Fe(CO)_nX]^+$ (n is 3 to 0). The most electronegative substituent, NO_3, labilises $[RC_3H_4Fe(CO)_nX]^+$ (n is 3 to 0), and hence its contribution to the overall fragmentation pattern is less and NO_3 elimination is predominant in this case. The least electronegative substituent, iodine, stabilises $[RC_3H_4\text{-}Fe(CO)_nX]^+$ significantly [which is also verified by the presence of intense molecular peaks in the spectra of (IV)], and thus the contribution of these ions towards the overall fragmentation pattern is significant while that of the ions formed through Fe–CO bond rupture decreases. The ratios φ_4/φ_1, φ_8/φ_4, φ_{12}/φ_8, and $\varphi_{16}/\varphi_{12}$ do not depend on the substituent X, an observation which may be explained by assuming that the ions φ_4, φ_8, and φ_{12} are not formed from the ions φ_5, φ_9, and φ_{14}, respectively. The ratio φ_4/φ_1 should not be influenced by the substituent X since the ions φ_4 are formed from φ_1 which contain no X. On the other hand, if it is assumed that φ_4 are formed from both φ_1 and φ_5, than the φ_4 portion formed from φ_5 will depend on the substituent X, which would make the ratio φ_4/φ_1 vary with X. The same is true for φ_8 and φ_{12}.

The φ_{11} ions result from the allyl–iron bond rupture in φ_{14} (as is shown by the presence of the corresponding metastable ions), the ratio $\varphi_{11}/\varphi_{14}$ increasing over the series $\mathrm{NO_3}<\mathrm{Cl}<\mathrm{Br}<\mathrm{I}$. Since the quantity $\varphi_{11}/\varphi_{14}$ is a characteristic of the allyl–iron bond strength and of the $[FeX]^+$ stability, it must be assumed that the allyl–iron bond strength falls over the series $\mathrm{I}<\mathrm{Br}<\mathrm{Cl}<\mathrm{NO_3}$ and/or the stability of the ions $[FeX]^+$ increases as this series is traversed. The presence of substituents in the allyl ligand also affects the fragmentation pattern, *e.g.*, peaks corresponding to iron-containing ions are predominant when R is H, CH_3, or Br whereas the fraction of iron-free ions rises noticeably when phenyl-substituted substrates are present. It is probable that in the latter case the charge is localised on the phenyl ring, which results in the formation of an intense $C_9H_9^+$ ion(m/e 117) which decomposes further to give $C_9H_8^+$ (m/e 116), $C_9H_7^+$ (m/e 115), $C_8H_7^+$ (m/e 103), $C_8H_6^+$ (m/e 102), $C_7H_7^+$ (m/e 91), $C_5H_5^+$ (m/e 65), $C_3H_3^+$ (m/e 39), $C_6H_6^+$ (m/e 78), $C_6H_5^+$ (m/e 77), and $C_4H_3^+$ (m/e 51). In addition to the ions $[C_6H_5C_3H_4Fe(CO)_n]^+$ (n is 1 or 2), phenylated compounds produce intense peaks of $[C_6H_5C_3H_5Fe(CO)_n]^+$. The latter peak is a maximum in the spectrum of (IIId) (R is 1-Ph, X is Br). The nature and the origin of these ions are as yet not clear.

The regularities reported here for the elimination of CO from allyliron halides are consistent with the behaviour of these halides in their reaction with triphenylphosphine[9]. A logical extension of this work could involve a stricter correlation between the mass-spectral results and the chemical behaviour of the compounds or, on the other hand, with their NMR data[10].

EXPERIMENTAL

Mass spectra were measured with a MX-1303 machine. The direct introduction system was maintained at 25 to 30°, the ionising chamber at 150°, and the ionising voltage was 50 V. Under these conditions, the substrates are thermally stable, their decomposition being apparent only when the temperature of the introduction system exceeded 50°.

The compounds $RC_3H_4Fe(CO)_3X$, where R is H, 1-Me, 1-Ph, 2-Br, 1-Me (X is Br), were synthesised by heating iron nonacarbonyl up to 45° with an equimolar amount of the respective allyl chloride or bromide in heptane for two hours. The compounds were purified by crystallisation from heptane at low temperatures. Compounds with R=H, 2-Me, 1-Me (X=I), or R=H, 2-Me, 1-Me, 1-Ph ($X=NO_3$) were synthesised through the metathesis of the chlorides or bromides with KI or $AgNO_3$ in acetone or methanol at room temperature[11]. The carbonyls (Ib) (R is 1-Me, X is NO_3), (Ic) (R is 2-Me, X is NO_3), (IIIc) (R is 2-Me, X is Br), (IIId) (R is 1-Ph, X is Br), and (IIIe) (R is 2-Ph, X is Br) are new compounds.

Preparation of 2-Me-π-$C_3H_4Fe(CO)_3NO_3$ *(Ic)*

2-Me-π-$C_3H_4Fe(CO)_3Cl$ (1.38 g, 0.006 mole), dissolved in 40 ml dry CH_3OH, was mixed with $AgNO_3$ (1.19 g, 0.007 mole) dissolved in 60 ml CH_3OH, in an argon atmosphere at ca. 20°. After 30 min, AgCl was filtered off, the filtrate evaporated *in vacuo*, and the residue re-crystallised from benzene/ligroin (3/1) to give the (2-methallyl)-iron tricarbonyl nitrate, 0.71 g (46%), as yellow plates. (Found: C, 32.88; H, 2.78; Fe, 21.68; N, 5.51. $C_7H_7FeNO_6$ calcd.: C, 32.71; H, 2.75; Fe, 21.73; N, 5.45%.)

Preparation of 1-Me-π-$C_3H_4Fe(CO)_3NO_3$ *(Ib)*

This was obtained in a similar fashion. $AgNO_3$ (0.68 g, 0.004 mole) and 1-Me-π-$C_3H_4Fe(CO)_3Cl$ (0.92 g, 0.004 mole) gave 0.29 g (28%) of the product as shining yellow plates, m.p. 92° (benzene/pentane, 3/1). (Found: C, 32.69; H, 2.76; Fe, 21.77; N, 5.36. $C_7H_7FeNO_6$ calcd.: C, 32.71; H, 2.75; Fe, 21.73; N, 5.45%.) This compound was found to be much less stable in solution than the product of the previous experiment.

Preparation of 2-Me-π-$C_3H_4Fe(CO)_3Br$ *(IIIc)*

2-Me-π-$C_3H_4Fe(CO)_3Cl$ (0.81 g, 0.0035 mole), dissolved in 10 ml abs. CH_3OH, was treated with two portions of $(CH_3)_4NBr$, each consisting of 0.54 g (0.005 mole) of the ammonium salt in 50 ml CH_3OH. The reaction mixture was stirred for 1½ h in an argon atmosphere at ca. 20°. The solvent was rapidly evaporated *in vacuo* and the residue re-crystallised from heptane to give (2-methylallyl)iron tricarbonyl bromide as brown needles, 0.28 g (29%), m.p. 98° (dec.). (Found: C, 30.63; H, 2.57; Br, 29.14; Fe, 20.36. $C_7H_7BrFeO_2$ calcd.: C, 30.59; H, 2.57; Br, 29.07; Fe, 20.32%.)

Preparation of 1-Ph-π-$C_3H_4Fe(CO)_3Br$ *(IIId)*

A mixture of $Fe_2(CO)_9$ (18.19 g, 0.05 mole) and cinnamyl bromide (9.85 g, 0.05 mole) was heated in 150 ml hexane to temperatures up to 40–45° for two hours in an argon atmosphere. The solvent was removed *in vacuo* and the residue repeatedly extracted with hexane followed by methylene chloride. The combined extracts were evaporated *in vacuo* and the residue re-crystallised from hexane to give (1-phenylallyl)-iron tricarbonyl bromide, 10.03 g (60 %), as brown needles, m.p. 101.5° (dec.) after three re-crystallisations from hexane. (Found: C, 42.93; H, 2.84; Br, 23.75; Fe, 16.61. $C_{12}H_9BrFeO_3$ calcd.: C, 42.77; H, 2.69; Br, 23.72; Fe, 16.57 %.)

Preparation of $2\text{-}Ph\text{-}\pi\text{-}C_3H_4Fe(CO)_3Br$ *(IIIe)*

This was prepared in a similar way. $Fe_2(CO)_9$ (6 g, 0.016 mole) and α-(bromomethyl)styrene (5.49 g of its mixture presumably containing 40 % 1-bromo-2-phenyl-1-propene) gave the compound, 0.22 g (4 %), as brown needles, m.p. 92° (dec.; from THF/hexane, 1/4). (Found: C, 42.77; H, 2.82; Br, 23.60; Fe, 16.56. $C_{12}H_9BrFeO_3$ calcd.: C, 42.77; H, 2.69; Br, 23.72; Fe, 16.57 %.)

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