Journal of Organometallic Chemistry
Elsevier Sequoia S.A., Lausanne
Printed in The Netherlands

ORGANOMETALLIC TRANSITION METAL DERIVATIVES CONTAINING FLUORINE

IV*. REACTIONS OF PERFLUOROALKYL DERIVATIVES OF CYCLOPENTADIENYLMETAL CARBONYLS OF IRON AND MOLYBDENUM WITH TERTIARY PHOSPHINES

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(Received June 19th, 1969)

SUMMARY

Ultraviolet irradiation of the perfluoroalkyliron derivatives $R_fFe(CO)_2C_5H_5$ [$R_f = C_2F_5$ and $(CF_3)_2CF$] with the saturated chelating ditertiary phosphine $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$(Pf–Pf) results in stepwise substitution of the carbonyl groups giving first the monodentate monometallic derivatives $R_fFe(CO)$(Pf–Pf)(C_5H_5) and then the carbonyl-free bidentate monometallic derivatives R_fFe(Pf–Pf)(C_5H_5). By contrast, UV irradiation of the same perfluoroalkyliron derivatives with the unsaturated chelating ditertiary phosphine *cis*-$(C_6H_5)_2PCH{=}CHP(C_6H_5)_2$ (*c*Pf=Pf) proceeds directly to the carbonyl-free bidentate monometallic derivatives R_fFe(*c*Pf=Pf)(C_5H_5) without formation of an isolable monodentate monometallic derivative. UV irradiation of the $R_fFe(CO)_2C_5H_5$ derivatives with triphenylphosphine gives the substitution products $R_fFe(CO)[P(C_6H_5)_3](C_5H_5)$ ($R_f = CF_3$, CF_3CO, C_2F_5, and $(CF_3)_2CF$); IR and NMR spectral data on the compound of this type with the relatively bulky $(CF_3)_2CF$ group suggest the presence of conformational isomers arising from hindered rotation about the iron–perfluoroalkyl bond. Similar UV irradiation of the molybdenum derivative $CF_3Mo(CO)_3C_5H_5$ with the appropriate tertiary phosphines gives the substitution products $CF_3Mo(CO)_2[P(C_6H_5)_3](C_5H_5)$ and $CF_3Mo(CO)$(Pf–Pf)(C_5H_5). An improved preparation of the $R_fFe(CO)_2C_5H_5$ [$R_f = C_2F_5$ and $(CF_3)_2CF$] derivatives from the corresponding perfluoroalkyliron tetracarbonyl iodides $R_fFe(CO)_4I$ and thallium(I) cyclopentadienide is described.

INTRODUCTION

The reactions of the alkyls $CH_3Fe(CO)_2C_5H_5$ and $CH_3Mo(CO)_3C_5H_5$ with tertiary phosphines have been shown to give acyl derivatives of the type $CH_3COFe(CO)(PR_3)(C_5H_5)$[2,3] and $CH_3COMo(CO)_2(PR_3)(C_5H_5)$[4,5], respectively. This paper

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* For Part III of this series see ref. 1.

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describes analogous reactions between perfluoroalkyls of the types $R_fFe(CO)_2C_5H_5$ and $R_fMo(CO)_3C_5H_5$[6] and tertiary phosphines. The chelating ditertiary phosphines $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$ and *cis*-$(C_6H_5)_2PCH{=}CHP(C_6H_5)_2$ were also included in this study in view of the interesting differences in the behavior of these two ligands upon reaction with the molybdenum alkyls $RMo(CO)_3C_5H_5$ (R = methyl or benzyl)[7]. The general preparative techniques in this study resembled those utilized in the recent study of the reaction between the silicon–iron and tin–iron derivatives of the type $(CH_3)_3EFe(CO)_2C_5H_5$ (E = Si or Sn) and tertiary phosphine ligands[8].

EXPERIMENTAL

The starting materials $[C_5H_5Fe(CO)_2]_2$[9], $CF_3Mo(CO)_3C_5H_5$[6], 1,2-bis(diphenylphosphino)ethane (Pf–Pf)[10], and *cis*-1,2-bis(diphenylphosphino)ethylene (*c*Pf=Pf)[11] were prepared by the previously published procedures. The modified procedures described below were used to prepare the perfluoroalkyl derivatives of cyclopentadienyliron dicarbonyl.

Preparation of $CF_3COFe(CO)_2C_5H_5$

A solution of 100 mmoles of $NaFe(CO)_2C_5H_5$ in 300 ml of tetrahydrofuran (redistilled over $LiAlH_4$) was treated at −78° with 21 g (100 mmoles) of redistilled trifluoroacetic anhydride. The reaction mixture was allowed to warm slowly to room temperature and then stirred for 16 h at room temperature. Solvent was then removed from the reaction mixture at ~25°/40 mm. The product was distilled out of the residue at 110°/0.02 mm. The crude product was purified by crystallization from pentane followed by sublimation at 60°/0.1 mm to give 6.3 g (23% yield) of $CF_3COFe(CO)_2C_5H_5$. The reported procedure[6] for the preparation of $CF_3COFe(CO)_2C_5H_5$ utilized the same reaction but was less convenient to carry out and gave a yield of only 9%.

The photochemical decarbonylation of $CF_3COFe(CO)_2C_5H_5$ to give $CF_3Fe(CO)_2C_5H_5$ was carried out as described previously[6]. As the scale was increased the yield of product dropped drastically. For this reason only relatively small quantities of $CF_3Fe(CO)_2C_5H_5$ were available for this study.

Preparation of $(CF_3)_2CFFe(CO)_2C_5H_5$

A mixture of 26 g (56 mmoles) of $(CF_3)_2CFFe(CO)_4I$[1], 17 g (61 mmoles) of thallium cyclopentadienide[12], and 250 ml of redistilled tetrahydrofuran was stirred for 6 h at room temperature. The original red color of the perfluoroalkyliron tetracarbonyl iodide changed almost immediately to orange-brown and a yellow precipitate of thallium(I) iodide separated. After the reaction period was over, the reaction mixture was filtered and solvent was removed from the filtrate at ~25°/40 mm. The residue was sublimed at 70–75°/0.1 mm to give 13.4 g (70% yield) of yellow-orange $(CF_3)_2CFFe(CO)_2C_5H_5$, m.p. 75–76°.

A similar procedure was used to prepare the pentafluoroethyl derivative $C_2F_5Fe(CO)_2C_5H_5$ from $C_2F_5Fe(CO)_4I$[13] and thallium cyclopentadienide. This approach was not used for the preparation of the trifluoromethyl derivative $CF_3Fe(CO)_2C_5H_5$ owing to difficulties in preparing sufficient quantities of the starting material $CF_3Fe(CO)_4I$[13].

Reactions of $R_fFe(CO)_2C_5H_5$ *and* $CF_3Mo(CO)_3C_5H_5$ *with tertiary phosphines*:

The following general procedures were used. Further details are given in Table 1.

Method A. Hexane solution. A hexane solution containing stoichiometric quantities of the $R_fFe(CO)_2C_5H_5$ or $R_fMo(CO)_3C_5H_5$ derivative and the tertiary phosphine in the 0.4 to 1.0 gram range was exposed to UV irradiation from an Englehard–Hanovia watercooled 450 watt lamp inserted into one neck of the reaction vessel. After the reaction period was over, the reaction mixture was filtered. The precipitate contained any $R_fFe(CO)(Diphos)(C_5H_5)$ derivative formed in the reaction. It was purified by recrystallization from a mixture of dichloromethane and hexane.

Solvent was removed from the filtrate at ~25°/40 mm. The solid remaining was the $R_fFe(CO)[P(C_6H_5)_3](C_5H_5)$ or $R_fFe(Diphos)(C_5H_5)$ derivative. It was purified by recrystallization from a mixture of dichloromethane and hexane.

Method B. Benzene solution. A benzene solution containing stoichiometric quantities of the $R_fFe(CO)_2C_5H_5$ or $R_fMo(CO)_3C_5H_5$ derivative and the tertiary phosphine in the 0.4 to 1.0 gram range was exposed to UV irradiation as described above for Method A. When using iron derivatives, the course of the reaction was followed by observing the disappearance of the ν(CO) frequencies from the IR spectrum of the reaction mixture. After the reaction period was over, solvent was removed from the reaction mixture at ~25°/40 mm. The residue was recrystallized from mixtures of dichloromethane and hexane to give the desired product.

DISCUSSION

The reactions of triphenylphosphine with the perfluoroalkyls $R_fFe(CO)_2C_5H_5$ and $CF_3Mo(CO)_3C_5H_5$ do not lead to insertion of a carbonyl group into the metal–perfluoroalkyl bond to give a perfluoroacyl derivative. Instead, one carbonyl group is lost to give the monosubstituted triphenylphosphine derivative of the perfluoroalkyl. This behavior differs from that previously observed for the methyl derivatives $CH_3Fe(CO)_2C_5H_5$[2,3] and $CH_3Mo(CO)_3C_5H_5$[4,5] which react with triphenylphosphine to give the acetyl derivatives $CH_3COFe(CO)[P(C_6H_5)_3](C_5H_5)$ and $CH_3COMo(CO)_2[P(C_6H_5)_3](C_5H_5)$, respectively. The inability for a carbonyl group to be inserted into a perfluoroalkyl–metal bond is a further consequence of the strengthening of a metal–alkyl bond by substitution of hydrogen atoms with fluorine atoms[14]. The stability of perfluoroacyl derivatives containing triphenylphosphine ligands is demonstrated by the preparation of the trifluoroacetyl derivative $CF_3COFe(CO)[P(C_6H_5)_3](C_5H_5)$ by UV irradiation of $CF_3COFe(CO)_2C_5H_5$ with triphenylphosphine; some $CF_3Fe(CO)[P(C_6H_5)_3](C_5H_5)$ is also produced in this reaction owing to photochemical decarbonylation[6] of $CF_3COFe(CO)_2C_5H_5$ to the trifluoromethyl derivative $CF_3Fe(CO)_2C_5H_5$ followed by reaction of this trifluoromethyl derivative with triphenylphosphine.

The photochemical reactions of the iron derivatives $R_fFe(CO)_2C_5H_5$ with the saturated chelating ditertiary phosphine 1,2-bis(diphenylphosphino)ethane, $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$(Pf–Pf) proceed according to the following two step sequence:

$$R_fFe(CO)_2C_5H_5 + \text{Pf–Pf} \rightarrow R_fFe(CO)(\text{Pf–Pf})(C_5H_5) + CO$$

$$R_fFe(CO)(\text{Pf–Pf})(C_5H_5) \rightarrow R_fFe(\text{Pf–Pf})(C_5H_5) + CO$$

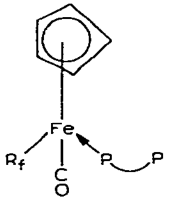
TABLE 1

NEW COMPOUNDS PREPARED IN THIS WORK

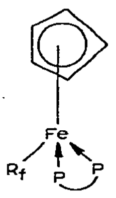
Compound[a]	Color	M.p.[b] (°C)	Preparation		Analyses found (calcd.) (%)[d]				Infrared ν(CO) (cm⁻¹)[e]
			Method[c]	Yield (%)	C	H	F	P	
$(CF_3)_2CFFe(CO)_2C_5H_5$[f]	Yellow	75–76°	TlC_5H_5	70	34.8 (34.7)	1.2 (1.4)			2046, 2003 [C_6H_{12}]
$CF_3COFe(CO)(PPh_3)(C_5H_5)$[g]	Yellow	180–181° (dec.)	A(4)[i]	34	61.1 (61.4)	4.0 (4.0)	11.3 (11.2)	6.2 (6.1)	1962, 1629 [C_6H_{12}]
$CF_3Fe(CO)(PPh_3)(C_5H_5)$[h]	Yellow	165–167°	A(12)	78	63.4 (62.5)	4.3 (4.2)	12.0 (11.9)	5.9 (6.4)	1968 [C_6H_{12}]
$C_2F_5Fe(CO)(PPh_3)(C_5H_5)$	Orange	143–144°	A(15)	44	58.9 (59.2)	4.1 (3.7)	17.9 (17.8)	6.1 (5.8)	1966 [C_6H_{12}]
$C_2F_5Fe(CO)(Pf\text{–}Pf)(C_5H_5)$	Orange	165° (dec.)	A(22)	70	59.3 (61.2)	4.2 (4.4)	15.0 (14.3)	9.9 (9.2)	1950 [CH_2Cl_2]
$C_2F_5Fe(Pf\text{–}Pf)(C_5H_5)$	Orange	159–160° (dec.)	B(75) A(22)	37 13	62.1 (62.1)	4.5 (4.6)	15.0 (14.9)	9.8 (9.7)	
$C_2F_5Fe(cPf{=}Pf)(C_5H_5)$	Red-brown	140–141°	A(21)	80	62.5 (62.3)	4.2 (4.3)	14.8 (14.9)	10.0 (9.7)	
$(CF_3)_2CFFe(CO)(PPh_3)(C_5H_5)$	Red	152–153°	A(13)	44	55.9 (55.4)	3.4 (3.4)	23.0 (22.6)	5.3 (4.9)	1978, 1963 [C_6H_{12}]
$(CF_3)_2CFFe(CO)(Pf\text{–}Pf)(C_5H_5)$	Orange	192° (dec.)	A(18)	52	58.4 (58.7)	4.2 (4.1)	19.1 (18.6)	8.9 (8.6)	1955 [CH_2Cl_2]
$(CF_3)_2CFFe(Pf\text{–}Pf)(C_5H_5)$	Red-brown	136°	B(40) A(18)	85 36	59.2 (59.3)	4.3 (4.2)	17.6 (19.3)	9.1 (9.0)	
$(CF_3)_2CFFe(cPf{=}Pf)(C_5H_5)$	Red-purple	140° (dec.)	A(12)	87	59.9 (59.5)	4.4 (4.0)	18.3 (19.4)	9.3 (9.0)	
$CF_3Mo(CO)_2(PPh_3)(C_5H_5)$	Orange	154–156° (dec.)	A(12)	63	56.9 (56.9)	3.7 (3.6)	10.1 (10.4)	10.0 (9.7)	1979, 1885
$CF_3Mo(CO)(Pf\text{–}Pf)(C_5H_5)$	Brown	215° (dec.)	B(40)	80	60.0 (60.4)	4.4 (4.0)	18.3 (19.4)	9.3 (9.0)	1848 [CH_2Cl_2]

[a] The following abbreviations are used in this Table: Ph = phenyl; Pf–Pf = 1,2-bis(diphenylphosphino)ethane; cPf=Pf = *cis*-1,2-bis(diphenylphosphino)ethylene. [b] These melting points were taken in capillaries and are uncorrected. [c] Methods A and B refer to the methods thus designated in the Experimental Section of the text. The reaction time in hours is given in parentheses. TlC_5H_5 refers to the use of thallium cyclopentadienide in tetrahydrofuran solution. [d] These analyses were performed by Pascher Mikroanalytisches Laboratorium, Bonn, Germany. [e] These spectra were taken in the indicated media and recorded on a Perkin–Elmer Model 621 spectrometer with grating optics. [f] Found: Fe, 15.9. Calcd.: Fe, 16.1. [g] Found: Fe, 10.8. Calcd.: Fe, 11.0. [h] Found: Fe, 11.8. Calcd.: Fe, 11.6. [i] In order to remove the $CF_3Fe(CO)[P(C_6H_5)_3](C_5H_5)$ concurrently formed in this reaction the crude product was chromatographed on an alumina column in a mixture of dichloromethane and hexane.

The intermediate monodentate monometallic[15] derivatives $R_fFe(CO)(Pf\text{–}Pf)(C_5H_5)$ (I) can be most readily isolated if the photochemical reactions are carried out in hexane solution. Under these conditions the monodentate monometallic products precipitate from the reaction mixture as they are formed thereby protecting them somewhat from further photochemical decarbonylation to give the bidentate monometallic derivatives $R_fFe(Pf\text{–}Pf)(C_5H_5)$ (II). The pure bidentate monometallic derivatives (II) are most readily obtained in the pure state by carrying out the photochemical reactions in benzene solution until the infrared spectrum of an aliquot of the solution indicates the disappearance of all $\nu(CO)$ frequencies.



(I)



(II)

Recent work[7,15] has shown the *unsaturated* chelating ditertiary phosphine *cis*-1,2-bis(diphenylphosphino)ethylene, $(C_6H_5)_2PCH{=}CHP(C_6H_5)_2$ (*c*Pf=Pf) to form chelate (bidentate monometallic) derivatives more readily than the saturated ditertiary phosphine $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$ discussed above. Photochemical reactions of the $R_fFe(CO)_2C_5H_5$ ($R_f{=}C_2F_5$ or $(CF_3)_2CF$) derivatives with the unsaturated ditertiary phosphine proceed directly to the bidentate monometallic derivatives $R_fFe(c\text{Pf=Pf})(C_5H_5)$ (II). No intermediate monodentate monometallic derivatives $R_fFe(CO)(c\text{Pf=Pf})(C_5H_5)$ (I) are isolated even when the photochemical reactions are carried out in hexane solution. A comparison of the behaviors of the saturated ditertiary phosphine $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$ and the unsaturated ditertiary phosphine *cis*-$(C_6H_5)_2PCH{=}CHP(C_6H_5)_2$ upon treatment with the iron derivatives $R_fFe(CO)_2C_5H_5$ [$R_f{=}C_2F_5$ or $(CF_3)_2CF$] provides a further indication of the greater chelating ability of the unsaturated ditertiary phosphine.

The IR spectra of the new carbonyl derivatives prepared in this work exhibit the expected number of $\nu(CO)$ frequencies except for $(CF_3)_2CFFe(CO)[P(C_6H_5)_2](C_5H_5)$ which exhibits two $\nu(CO)$ frequencies in cyclohexane solution possibly arising from conformational isomers similar to those observed by Jetz and Graham[16] for certain silyliron derivatives of the type $R_2R'SiFe(CO)_2C_5H_5$. As usual, increasing substitution of carbonyl groups with tertiary phosphines lowers the $\nu(CO)$ frequencies owing to increased population of the antibonding orbitals from increased metal–carbon retrodative bonding. The proton NMR spectra of the new compounds exhibit the expected resonances from the π-cyclopentadienyl rings as well as resonances from the ligand protons. Substitution of one carbonyl group in an $R_fFe(CO)_2C_5H_5$ derivative with a tertiary phosphine raises the chemical shift of the π-C_5H_5 resonance by about 0.5 ppm. However, substitution of the second carbonyl group with a tertiary phosphine has only a small additional effect on the chemical shift of the π-C_5H_5 resonance. A similar "saturation" effect has been noted in the tertiary phosphine substitution products of the $(CH_3)_3EFe(CO)_2C_5H_5$($E{=}Si$ or Sn) derivatives[8]. In some of the better spectra the phosphorus atoms of the $(C_6H_5)_3P$ ligand were observed to split the π-C_5H_5 or the CF_3 resonances by about 1.5 Hz. However, such

TABLE 2

NMR SPECTRA OF SOME NEW COMPOUNDS PREPARED IN THIS WORK

Compound[a]	^{19}F NMR[b,c] (ϕ)				^{1}H NMR[c,d] (τ)	
	Solvent	ϕ(CF)	ϕ(CF$_2$)	ϕ(CF$_3$)	Solvent	τ(C$_5$H$_5$)
A. CF_3 derivatives						
$CF_3COFe(CO)(PPh_3)(C_5H_5)$	CH_2Cl_2			79.3 s	$CDCl_3$	5.50 d (1.5)
$CF_3Fe(CO)(PPh_3)(C_5H_5)$	CH_2Cl_2			−13.7 d (1.6)	$CDCl_3$	5.49
$CF_3Fe(CO)_2C_5H_5$[e]	CH_2Cl_2			−11.6 s	$CHCl_3$	5.06
$CF_3Mo(CO)_2(PPh_3)(C_5H_5)$	CH_2Cl_2			−11.3 d (1.5)		
$CF_3Mo(CO)_3C_5H_5$	CH_2Cl_2			−11.9 s		
B. C_2F_5 derivatives						
$C_2F_5Fe(CO)(PPh_3)(C_5H_5)$	CH_2Cl_2		65.0	81.7		
$C_2F_5Fe(CO)$(Pf–Pf)(C_5H_5)	CH_2Cl_2		65.8	81.5	$(CD_3)_2CO$	5.60 s
C_2F_5Fe(Pf–Pf)(C_5H_5)	CH_2Cl_2		63.5	79.7	$CDCl_3$	5.67 s
C_2F_5Fe(*c*Pf=Pf)(C_5H_5)	CH_2Cl_2		66.4	80.3		
$C_2F_5Fe(CO)_2C_5H_5$	CH_2Cl_2		62.8	82.1		
C. $(CF_3)_2CF$ derivatives						
$(CF_3)_2CFFe(CO)(PPh_3)(C_5H_5)$	CH_2Cl_2	162.9?		66.9 sp (6)	$CDCl_3$	5.47 s
$(CF_3)_2CFFe(CO)$(Pf–Pf)(C_5H_5)	CH_2Cl_2	[f]		65.0		
$(CF_3)_2CFFe$(Pf–Pf)(C_5H_5)	CH_2Cl_2	[f]		65.1 d (12)	$CDCl_3$	5.67 s
$(CF_3)_2CFFe$(*c*Pf=Pf)(C_5H_5)	CH_2Cl_2	[f]		64.0 b		
$(CF_3)_2CFFe(CO)_2C_5H_5$	CH_2Cl_2	164.7 qn (12)		68.5 d (12)	$(CH_3)_2CO$	4.80 s

[a] Ph = phenyl; Pf–Pf = 1,2-bis(diphenylphosphino)ethane; *c*Pf=Pf = cis-1,2-bis(diphenylphosphino)ethylene. [b] These ^{19}F NMR spectra were taken in the indicated solvents and recorded on a Perkin–Elmer Hitachi R-20 spectrometer. Chemical shifts were determined using $CFCl_3$ (ϕ 0.0) or $CFCl_2CFCl_2$ (ϕ 67.8) as an internal standard. The scale used here was introduced by G. Filipovich and G. V. D. Tiers[17]. [c] The observed fine structure was indicated by the following abbreviations: s = singlet, d = doublet, qn = apparent quintet, sp = apparent septet, b = broad. Coupling constants in Hz are given in parentheses. [d] These ^{1}H NMR spectra were taken in the indicated solvents and recorded on a Varian HA-100 spectrometer using tetramethylsilane as an internal standard. [e] Data from R. B. King and M. B. Bisnette[18]. [f] Not observed.

splitting of the CF_3 resonances by phosphorus atoms was only observed in compounds where the CF_3 group is bonded directly to the metal atom. Thus a doublet CF_3 resonance was observed in the trifluoromethyl derivative $CF_3Fe(CO)[P(C_6H_5)_3](C_5H_5)$ but not in the trifluoroacetyl derivative $CF_3COFe(CO)[P(C_6H_5)_3](C_5H_5)$.

ACKNOWLEDGEMENT

We are indebted to the National Institute of General Medical Sciences for partial support of this work under Grants GM-14664-01 and GM-14664-02.

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