Reactions of a Terminal Phosphido-group in an Organoiron Complex. Part II.† New Routes to Derivatives of the Tricarbonyl(η-cyclopentadienyl)iron Cation

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Two new routes for the preparation of derivatives of $[(cp)Fe(CO)_3]^+$ (cp = η-cyclopentadienyl) are reported. Reaction of $[(cp)Fe(CO)_2\{P(CF_3)_2\}]$, (I), with excess of X_2 (X = Cl, Br, or I) gives $[(cp)Fe(CO)_2\{P(CF_3)_2X\}]$-$[X_3]$, (II), in quantitative yields. The tri-iodide salt is stable at room temperature but the tribromide and trichloride decompose to $[(cp)Fe(CO)_2X]$ and $(F_3C)_2PX$. With ICl, (II; X = Cl) is formed. A second method for the preparation of cationic phosphine complexes is protonation of (I) to give $[(cp)Fe(CO)_2\{(CF_3)_2PH\}]^+$, (IV), and of $[(cp)Fe(CO)_2\{P(E)(CF_2)_2\}]$, (III), to give $[(cp)Fe(CO)_2\{P(CF_3)_2(EH)\}]^+$ (V; E = O, S, or Se).

CATIONIC complexes of the form $[(cp)Fe(CO)_2PR_3]^+$ (cp = η-cyclopentadienyl) have been known for several years.[1-4] Early methods of preparation required high-pressure conditions to isolate $[(cp)Fe(CO)_3]^+$ which was then allowed to react with the phosphine PR_3. Recently, oxidation of $[\{(cp)Fe(CO)_2\}_2]$ by electrochemical means or by the action of iron(III) perchlorate or silver(I) salts in acetone solution provided the acetone complex $[(cp)Fe(CO)_2(OCMe_2)]^+$ which reacts readily with phosphines to yield the cationic complex.[5-8] We now report a simple high-yield route to the analogous cations $[(cp)Fe(CO)_2\{P(CF_3)_2X\}]^+$ (X = Cl, Br, or I). The starting material is the phosphido-complex $[(cp)Fe(CO)_2\{P(CF_3)_2\}]$, (I), which is prepared by the action of tetrakis(trifluoromethyl)diphosphane on $[\{(cp)Fe(CO)_2\}_2]$.[9]

RESULTS AND DISCUSSION

When (I) was allowed to react with an excess of halogen, 2 moles were consumed. No carbon monoxide was liberated, and excess of the free halogen was easily pumped off in the case of iodine or bromine. The chlorine product is unstable in the solid state, as discussed below.

The ^1H n.m.r. spectra of the products showed doublets for the resonance of the cp ring with a pronounced shift to low field compared to that of (I), showing that phosphorus is still attached to iron although the electron density around the ring hydrogen atoms must be lower. The ^{19}F n.m.r. spectra consisted of a simple doublet arising from F–P coupling. Very intense u.v. absorption bands of the solutions at 269 nm (X = Br) and 291 and 360 nm (X = I) can be assigned to Br_3^- and I_3^- respectively. The products are 1 : 1 conductors in acetonitrile solution[10] and can therefore be formulated as $[(cp)Fe(CO)_2\{P(CF_3)_2X\}][X_3]$, (II).

When the solvent was pumped off the product of the reaction of (I) with excess of chlorine, some decomposi-

tion was noted and the products were $[(cp)Fe(CO)_2Cl]$, (II; X = Cl), and $(F_3C)_2PCl$. The products suggest that the salt is decomposing by nucleophilic attack of free chloride ion [equation (1)], the source of the chloride

$$(II; X = Cl) + Cl^- \longrightarrow$$
$$[(cp)Fe(CO)_2Cl] + (F_3C)_2PCl \quad (1)$$
$$Cl_3^- \rightleftharpoons Cl^- + Cl_2 \quad (2)$$
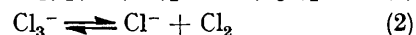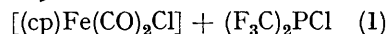
ion being Cl_3^-. This mode of decomposition is analogous to that reported[11] for the salt $[(cp)Ni(PBu_3)_2]Cl$ in benzene solution. In the presence of a small excess of chlorine over the 2 : 1 mole ratio, the acetonitrile solution was stable, demonstrating the suppression of equilibrium (2). Addition of $SnCl_4$ gave a stable solution from which the 1 : 1 electrolyte $[(cp)Fe(CO)_2\{P(CF_3)_2Cl\}][SnCl_5]$ was isolated.

The tri-iodide salt (II; X = I) was indefinitely stable at room temperature in the solid state and in solution. The tribromide was intermediate in stability between the tri-iodide and trichloride. After 1 week in acetonitrile solution, the salt (II; X = Br) decomposed by *ca.* 25% to $[(cp)Fe(CO)_2Br]$, $(F_3C)_2PBr$, and bromine; under the same conditions the trichloride completely decomposed. The ease of decomposition of the three salts thus follows the order of stability constants of the X_3^- ions in acetonitrile solution.[12] Photolysis of (II; X = Br) in acetonitrile solution gave free $(F_3C)_2PBr$.

With iodine monochloride, (I) absorbed 2 mol of reactant forming (II; X = Cl) in solution, presumably associated with I_2Cl^-. Breakdown occurred slowly at room temperature by chloride-ion attack to give $[(cp)Fe(CO)_2Cl]$. The reaction of (I) with iodine (2 mol) in the presence of Et_4NCl (1 mol) gave (II; X = Cl).

The observation of both halogen exchange at phosphorus and Fe–P bond cleavage suggests that halide ion can attack either at phosphorus or iron depending on the nature of the reagents. Another possibility is the initial

† Part I is ref. 9.

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formation of a neutral P^V complex which could decompose to give the products resulting from exchange or cleavage. In other experiments evidence was sought for a 1 : 1 adduct between (I) and bromine or iodine. With bromine (1 mol), (I) gave $[(cp)Fe(CO)_2Br]$ and $(F_3C)_2PBr$. The reaction of equimolar amounts of (I) and the salt (II; X = Br) gave the same products. In both cases, the reaction is probably initiated by the action of Br_2 on (I), giving free bromide ion which then rapidly attacks the iron atom of the cation. With iodine (1 mol), (I) gave a 1 : 1 adduct which slowly decomposed in acetonitrile solution; isolation of a pure complex was not achieved.

On treatment with water (II; X = Br) rapidly formed $[(cp)Fe(CO)_2\{P(O)(CF_3)_2\}]$, whereas $[(cp)Fe(CO)_2\{P(CF_3)_2Cl\}][SnCl_5]$ gave a new product which was thought to be $[(cp)Fe(CO)_2\{P(CF_3)_2(OH)\}]^+$, retaining the cationic structure. This suggested experiments to protonate the complexes (I) and $[(cp)Fe(CO)_2\{P(E)-(CF_3)_2\}]$ (III; E = O, S, or Se) in another approach to the synthesis of phosphine derivatives of $[(cp)Fe(CO)_3]^+$. Complex (I) did not react with anhydrous HCl or HI at room temperature in either polar or non-polar solvents;

pared both to that of the free ligand (τ 5·45) and neutral complexes of $(F_3C)PH$. The order of addition of the reagents and solvent used in the protonation of (I) by HCl and $SnCl_4$ is critical. Addition of excess of $SnCl_4$ to a solution of (I) in CCl_3F gave a yellow precipitate of a 1 : 1 adduct, not attacked subsequently by HCl, which is soluble in acetonitrile, the solution showing only the n.m.r. signals of (I). On the other hand, addition of $SnCl_4$ to (I) in nitromethane gave pale yellow crystals corresponding to a 2 : 1 reaction. Again HCl did not affect the product. On treating the solid with acetonitrile, (II; X = Cl) was formed in solution and a white solid, believed to be $SnCl_2$, remained.

Protonation of the P^V complexes, $[(cp)Fe(CO)_2-\{P(E)(CF_3)_2\}]$ (III; E = O, S, or Se) also succeeded when a mixture of HCl and $SnCl_4$ was used, although the point of attachment of the proton was the Group 6 atom so that the cations $[(cp)Fe(CO)_2\{P(CF_3)_2(EH)\}]^+$, (V), were formed. The evidence for the formulation of (V) is mainly from the n.m.r. spectra. The low-field cp resonance was similar to that of complexes (II) and the doublet signal in the ^{19}F n.m.r. spectrum slightly above that of the corresponding free ligand for (V; E = O or S).

Physical properties of some fluorocarbon–phosphorus compounds

Compound	$\tau(cp)$	$^3J(PH)/Hz$	δ [a]/p.p.m.	$^2J(PF)/Hz$	Λ/S cm^2 mol^{-1} (c/M)	Solvent
$[(cp)Fe(CO)_2\{P(CF_3)_2Cl\}][Cl_3]$ (II)	4·02	1·5	62·4	96	121·9 [b] (10^{-3})	MeCN
$[(cp)Fe(CO)_2\{P(CF_3)_2Br\}][Br_3]$ (II)	4·07	2·0	61·5	92	63 (6 × 10^{-2})	MeCN
$[(cp)Fe(CO)_2\{P(CF_3)_2I\}][I_3]$ (II)	4·26	2·0	58·4	78	82·9 [a] (8·5 × 10^{-3})	MeCN
$[(cp)Fe(CO)_2\{P(CF_3)_2H\}]^+$ (IV) [c]	4·11	1·9	52·6	79	48·8 (1·6 × 10^{-2})	MeNO$_2$
$[(cp)Fe(CO)_2\{P(CF_3)_2(OH)\}]^+$ (V)	4·20	1·5	67·3	85		MeNO$_2$
$[(cp)Fe(CO)_2\{P(CF_3)_2(SH)\}]^+$ (V)	4·10	b,s	61·4	86		MeNO$_2$
$[(cp)Fe(CO)_2\{P(CF_3)_2(SeH)\}]^+$ (V)	4·15	b,s	59·3	71		MeNO$_2$
$(F_3C)_2PCl$			60·7	82		MeCN
$(F_3C)_2PBr$			58·1	74		MeCN
$(F_3C)_2PI$			54·7	70		MeCN
$(F_3C)_2PH$ [d]			47·5	69		CCl$_3F$
$(F_3C)_2POH$			66·4	82		MeCN
$(F_3C)_2PSH$ [e]			59·6	77		CCl$_3F$

b,s = Broad singlet (usually <10 Hz half width).
[a] From CCl_3F. [b] Measured in excess of halogen. [c] $\tau(H) = 2·28 \times ^1J(PH) = 441$ Hz; $^3J(FH)$ 6·5 Hz. [d] Data from K. J. Packer, *J. Chem. Soc.*, 1963, 960. [e] Data from R. G. Cavell and H. J. Eméleus, *J. Chem. Soc.*, 1964, 5825.

at higher temperature, the Fe–P bond was cleaved and $(F_3C)_2PH$ liberated. A mixture of $SnCl_4$ and HCl, however, which acts as a stronger acid, protonated the complex to give $[(cp)Fe(CO)_2\{P(CF_3)_2H\}][SnCl_5]$, (IV). The product precipitated from CH_2Cl_2 but dissolved readily in nitromethane to give a conducting solution. The ^{19}F n.m.r. spectrum of the solution at 293 K gave a doublet, each line being broad (width at half height, 12 Hz). On cooling, the lines sharpened and at 253 K a doublet of doublets was fully resolved. When (IV) was prepared from DCl, the low-temperature spectrum consisted of a sharp doublet. These experiments suggest the presence of a $(F_3C)_2PH$ ligand. This was confirmed by the ^1H n.m.r. spectrum which showed at 293 K a sharp doublet at τ 4·11 for the cp resonance. At 253 K in CD_3NO_2, an additional doublet of septets, intensity 1/5 of that of the cp resonance, was detected. The n.m.r. parameters are given in the Table. The temperature variation of the spectrum is caused by fast exchange of H^+. The low-field chemical shift (τ 2·28) of the hydrogen on phosphorus indicates its much greater acidity com-

The single hydrogen atom was not located in the n.m.r. experiments carried out between 293 and 253 K, the effective lower limit of the solvent. Attempts to isolate (V; E = O or Se) in the solid state proved unsuccessful. A solution of (V; E = O) lost HCl on evaporation *in vacuo* and the resulting pale yellow solid was characterised as $[(cp)Fe(CO)_2\{P(CF_3)_2(O)\}]_2SnCl_4$. The protonation of (III; E = Se) was always accompanied by a deposit of red selenium, and (IV) was seen in the n.m.r. spectrum. Recent attempts to prepare $(F_3C)_2P(SeH)$ from $[(F_3C)_2-P]_2Se$ were unsuccessful, the products always being $(F_3C)_2PH$ and selenium. The results of these protonation experiments suggest an enhancement of the stability of $(F_3C)_2P(SeH)$ on co-ordination. A study of the n.m.r. spectra of (III; E = O or S) in H_2SO_4 showed almost identical results to those obtained with $HCl–SnCl_4$. No protonation of the metal atom occurred, as observed previously with iron complexes in H_2SO_4;[13] (III; E = Se) deposited red selenium in H_2SO_4.

[13] A. Davison, W. McFarlane, L. Pratt, and G. Wilkinson, *J. Chem. Soc.*, 1962, 3653.

In view of the strong electron-withdrawing character of the CF_3 group, the preparation of cations containing trifluoromethyl-substituted phosphorus atoms is not readily achieved. On the basis of conductivity measurements, Eméleus and Harris proposed that $(F_3C)_3PCl_2$ ionised in acetonitrile to give $(F_3C)_3PCl^+$ and $(F_3C)_3PCl_3^-$ ions.[14] The cations $[(cp)Fe(CO)_2\{P(CF_3)_2X\}]^+$ (X = I, Br, Cl, H, OH, SH, and SeH) appear to be the only other reported examples. Very recently, however, others have reported [15] the isolation from the reaction of $(F_3C)_2SbI$ with $[\{(cp)Fe(CO)_2\}_2]$ of a salt containing the related cation $[(cp)(OC)_2Fe\{\mu\text{-}Sb(CF_3)_2\}Fe(CO)_2(cp)]^+$. Clearly the presence of an amphoteric metal centre and ligands with donor–acceptor character can lead to more effective charge delocalisation and hence to stabilisation of the cation.

EXPERIMENTAL

Volatile compounds were handled in a Pyrex vacuum system by standard techniques. I.r. spectra were recorded on a Perkin-Elmer 457 spectrometer and u.v. spectra on a Perkin-Elmer 402 spectrometer. N.m.r. spectra were recorded either on a Perkin-Elmer R10 for ^1H nuclei or on a Bruker Spectrospin HFX operating at 90·00 MHz for ^1H and 84·66 MHz for ^{19}F nuclei, with MeCN (taken as τ 8·00) and CCl_3F as internal standards. Many of the reactions were carried out in n.m.r. tubes joined directly to Rotaflo taps so that they could be plugged into the vacuum line. Samples for n.m.r. spectroscopy were then made up in dry degassed solvents and sealed *in vacuo*. The complex $[(cp)Fe(CO)_2\{P(CF_3)_2\}]$, (I), was prepared as previously described.[9] Other reagents were carefully purified before use.

A conductivity cell was specially designed to allow the complexes to be prepared and the conductivities measured *in situ*. Platinum electrodes were used and the cell constant was determined using 0·01M-KCl solution.* The conductivities were measured using the usual type of Wheatstone-bridge network.

Reactions.—Complex (I) *with chlorine.* Chlorine (0·015 g; 0·21 mmol) reacted with complex (I) (0·029 g; 0·083 mmol) in dry CCl_3F, rapidly depositing a flocculent pale yellow precipitate. Removal of the volatile products left a yellow solid (0·034 g). On dissolving this product in dry acetonitrile the solution quickly turned dark red-brown. The ^{19}F n.m.r. spectrum confirmed the presence of $(F_3C)_2PCl$ and $[(cp)Fe(CO)_2\{P(CF_3)_2Cl\}]^+$, (II). The ^1H n.m.r. spectrum showed the presence of $[(cp)Fe(CO)_2Cl]$ and a small amount of unreacted starting material.

In another experiment (I) (0·040 g; 0·115 mmol) was allowed to react with excess of Cl_2 (0·026 g; 0·37 mmol) in dry MeCN solvent. Excess of Cl_2 and solvent were not removed; the n.m.r. spectra now showed only $[(cp)Fe(CO)_2\{P(CF_3)_2Cl\}][Cl_3]$, (II; X = Cl).

Complex (I) *with bromine.* Complex (I) (0·114 g; 0·32 mmol) and Br_2 (0·216 g; 1·35 mmol) reacted rapidly together in CCl_3F as solvent to produce a yellow precipitate. The volatile products were taken off *in vacuo* leaving the yellow solid, (II; X = Br), m.p. 107—109 °C (0·219 g; 0·32 mmol; 100% yield) (Found: C, 16·25; H, 0·75; Br, 48·0; Fe, 8·4%. Calc. for $C_9H_5Br_4F_6FeO_2P$: C, 16·35; H, 0·80; Br, 47·9; Fe, 8·60%), $\nu(CO)$ at 2 080 and 2 042 cm^{-1} in CH_2Cl_2 solution.

Complex (I) *with iodine.* Iodine (1·33 g; 5·24 mmol) and (I) (0·166 g; 0·48 mmol) were allowed to react together at

room temperature in dry CCl_3F solvent. On removal of the solvent and excess of iodine the brick red *solid* (II; X = I) (0·391 g, 0·46 mmol), m.p. 83·5—86 °C, $\nu(CO)$ at 2 090 and 2 058 cm^{-1} in CH_2Cl_2 solution, was isolated (Found: C, 12·6; H, 0·70; F, 13·5; I, 59·3. Calc. for $C_9H_5F_6I_4O_2P$: C, 12·65; H, 0·60; F, 13·35; I, 59·45%).

Complex (I) *with iodine monochloride.* Complex (I) (0·029 g; 0·085 mmol) was allowed to react at room temperature with ICl (0·255 g; 1·57 mmol) in dry CCl_3F solvent to produce (II; X = Cl), the cation being identified by n.m.r. spectroscopy.

Complex (I) *with* Cl_2–$SnCl_4$. Complex (I) (0·022 g; 0·064 mmol) and $SnCl_4$ (0·210 g, 0·81 mmol) were allowed to react at room temperature in $MeNO_2$ solution. Chlorine (0·105 g, 1·48 mmol) was distilled in to the tube and the reaction allowed to proceed. The product was soluble producing a pale yellow solution. Volatile products and solvent were then pumped off leaving $[(cp)Fe(CO)_2\{P(CF_3)_2Cl\}][SnCl_5]$ as a pale yellow *solid* (Found: Cl, 31·05; F, 16·55; Fe, 8·50; Sn, 17·7%. Calc. for $C_9H_5Cl_6F_6FeO_2PSn$: Cl, 31·4; F, 16·85; Fe, 8·25; Sn, 17·5%).

Chloride ion with (II; X = I). Dry Et_4NCl (0·011 g, 0·066 mmol) and (II; X = I) (0·054 g; 0·064 mmol) were allowed to react in acetonitrile as solvent. The cation (II; X = Cl) was identified by n.m.r. spectroscopy.

Complex (I) *with* (II; X = Br). The salt (II; X = Br) (0·061 g; 0·083 mmol) and (I) (0·033 g; 0·096 mmol) were allowed to react at room temperature in acetonitrile as solvent. The solution rapidly turned brown and $[(cp)Fe(CO)_2Br]$ and $(F_3C)_2PBr$ were identified by n.m.r. spectroscopy as the only products.

Complex (I) *with* HCl–$SnCl_4$. Complex (I) (0·035 g; 0·10 mmol) in CH_2Cl_2 was allowed to react with HCl (0·22 mmol) and $SnCl_4$ (0·22 mmol). A pale yellow precipitate rapidly formed. The volatile products and solvent were pumped off leaving a pale yellow *solid* $[(cp)Fe(CO)_2\{P(CF_3)_2H\}][SnCl_5]$, (IV) (0·065 g; 0·10 mmol; 100% yield) (Found: C, 16·6; H, 1·10; Cl, 27·3; Fe, 8·55. Calc. for $C_9H_6Cl_5F_6FeO_2PSn$: C, 16·8; H, 0·95; Cl, 27·6; Fe, 8·7%).

$[(cp)Fe(CO)_2\{P(O)(CF_3)_2\}]$, (III; E = O), *with* HCl–$SnCl_4$. Hydrogen chloride (0·30 mmol) and $SnCl_4$ (0·25 mmol) were condensed in to a tube containing (III; E = O) (0·066 g, 0·183 mmol) dissolved in $MeNO_2$. The resulting very pale yellow solution was transferred to the side-arm n.m.r. tube and $[(cp)Fe(CO)_2\{P(CF_3)_2(OH)\}]^+$, (V; E = O), was identified by n.m.r. spectroscopy. When this reaction was repeated and the volatiles removed, all the HCl was recovered and a pale yellow *solid*, which analysed for $[(cp)Fe(CO)_2\{P(O)(CF_3)_2\}]_2SnCl_4$ was obtained (Found: C, 21·7; H, 1·15; Cl, 14·4; Fe, 11·05; Sn, 12·3. Calc. for $C_{18}H_{10}Cl_4F_{12}Fe_2O_6P_2Sn$: C, 21·95; H, 1·00; Cl, 14·4; Fe, 11·35; Sn, 12·05%).

$[(cp)Fe(CO)_2\{P(S)(CF_3)_2\}]$, (III; E = S), *with* HCl–$SnCl_4$. The sulphur-containing *complex* $[(cp)Fe(CO)_2\{P(CF_3)_2SH\}][SnCl_5]$, (V; E = S) (Found: Fe, 8·40; Sn, 17·95. Calc. for $C_9H_6Cl_5F_6FeO_2PSSn$: Fe, 8·30; Sn, 17·57%) was prepared in a similar way by the action of HCl–$SnCl_4$ on (III; E = S).

$[(cp)Fe(CO)_2\{P(Se)(CF_3)_2\}]$, (III; E = Se), *with* HCl–$SnCl_4$. When a solution of (III; E = Se) in nitromethane was allowed to warm to room temperature with a slight

* $1M$ = 1 mol dm^{-3}.

14 H. J. Eméleus and G. S. Harris, *J. Chem. Soc.*, 1959, 1494.
15 W. R. Cullen, D. J. Patmore, and J. R. Sams, *Inorg. Chem.*, 1973, 12, 867.

molar excess of HCl–SnCl$_4$, a fine red precipitate of selenium was formed. N.m.r. spectra of the filtered solution showed the presence of (IV) in addition to another species which appeared from the n.m.r. parameters (see Table) to be [(cp)Fe(CO)$_2${P(CF$_3$)$_2$(SeH)}]$^+$, (V; E = Se).

Complex (III) *with* H$_2$SO$_4$. Complex (III; E = O) (0·051 g; 0·14 mmol) was dissolved in 98% H$_2$SO$_4$ (*ca.* 1 cm^3) in an n.m.r. tube. Me$_2$SO$_4$ and CF$_3$CO$_2$H were added as n.m.r. standards and the tube sealed *in vacuo*. The product was identified by n.m.r. spectroscopy as (V; E = O). The process was repeated for (III; E = S).

Photolytic Reactions.—After irradiation of a solution of (II; X = Br) in MeCN for 3·5 h through a Pyrex filter, the only detectable fluorine-containing product was (F$_3$C)$_2$PBr. Photolysis of [(cp)Fe(CO)$_2${P(CF$_3$)$_2$(OH)}][SnCl$_5$] in MeCN solution for 1 h through a Pyrex filter gave (F$_3$C)$_2$POH, in addition to the starting salt, as the only fluorine-containing product.