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THE REACTION OF FORMYL FLUORIDE WITH TRANSITION METAL COMPLEXES

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Summary

Formyl fluoride reacts with metal carbonyl anions in a manner similar to acetic formic anhydride. Although formyl complexes may have been formed as unstable intermediates, no neutral formyl complexes could be isolated but rather the expected decomposition products, the metal carbonyl hydrides or dimers, were produced. The attempted oxidative addition of formyl fluoride to various coordinately unsaturated metal complexes also did not result in the formation of formyl derivatives. HF adducts were obtained from the reaction of $Ir(CO)Cl(PR_3)_2$ or $M(PPh_3)_4$ (M = Pt or Pd) with formyl fluoride whereas $Ru(NO)Cl(PPh_3)_2$ and $Rh(PPh_3)_3Cl$ give the CO complexes $Ru(NO)(CO)Cl(PPh_3)_2$ and $Rh(CO)Cl(PPh_3)_2$, respectively.

Introduction

Formyl fluoride, the only isolable formyl halide, is a simple but interesting and reactive molecule. It is bifunctional containing both an acid halide and a reactive formyl group and reactivity may be expected from either of these sites. Thus far however, there seems to be no reports of the reaction of formyl fluoride with transition metal complexes. Of particular interest to us was the possibility of forming stable, neutral formyl complexes either by the reaction of transition metal carbonyl anions with formyl fluoride or by the oxidative addition of formyl fluoride to coordinately unsaturated transition metal complexes. Only a few stable neutral formyl complexes are known such as $Os(CO)_2(PPh_3)_2H(CHO)$ prepared by the oxidative addition of formaldehyde to $Os(CO)_2(PPh_3)_3$ through an intermediate η^2-formaldehyde complex [1], $Os(CO)_2(PPh_3)_2Cl(CHO)$ which was prepared by an indirect route [2] and $(C_5H_5)Re(NO)(CHO)(PPh_3)$ prepared by the hydride reduction of $(C_5H_5)Re(CO)(NO)(PPh_3)^+$ [3,4].

The oxidative addition of acyl halides is a well known reaction [5], although little work seems to have been carried out with the less well known fluorides.

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As an alternative to oxidative addition across the C—F bond formyl fluoride is also capable of reaction across the C—H bond. Reactions of this sort are not common but have been implicated in the decarbonylation of aldehydes by various rhodium complexes [6] and in the recently prepared osmium formyl [1] and acetyl [7] complexes derived from the corresponding aldehydes. By isolating the hydridoacyl intermediate in the decarbonylation of 8-quinolinecarboxaldehyde by $Rh(PPh_3)_3Cl$, Suggs has shown the oxidative addition of the C—H bond of the aldehyde to be a key step in the reaction [8].

Experimental

General

All reagents were obtained from commercial sources if possible or were prepared by standard literature methods. The most convenient method of preparation of formyl fluoride was from potassium hydrogen fluoride, anhydrous formic acid and benzoyl chloride [9]. After careful redistillation, a pure material is obtained whose spectrum is identical to that described [10]. If stored at low temperatures in teflon containers, no decomposition was observed after several days, however, more rapid decomposition takes place at ambient temperatures in contact with glass or especially stainless steel.

Solvents were generally distilled from $LiAlH_4$ and stored over sodium in a nitrogen filled dry box.

Elemental analyses were carried out by the Analytical and Information Division, Exxon Research and Engineering Company, and by Galbraith Laboratories, Inc., Knoxville, Tennessee. Infrared spectra were recorded on a Perkin-Elmer Model 283 spectrophotomer and NMR spectra obtained on a Varian A-60 or CRT-20 instrument.

All manipulations were carried out in a nitrogen-filled glove box.

Reactions

Reactions of metal carbonyl anions with formyl fluoride

Solutions of various metal carbonyl anions including $Mn(CO)_5^-$, $CpMo(CO)_3^-$, $Co(CO)_4^-$, $CpFe(CO)_2^-$ or $CpW(CO)_3^-$ were prepared by sodium amalgam reductions of the corresponding dimers, filtered and adjusted such that the concentration of the anion was approximately 20 millimoles in 100 ml THF. The solutions were cooled to —10°C and 0.96 g (20 mmol) HCOF was added to each solution with stirring. A grey precipitate of NaF (identified and determined semiquantitatively by reaction with aqueous $CaCl_2$) formed in all cases with some gas evolution. After stirring for 1—2 minutes, the solutions were filtered on a medium frit and the filtrates analyzed by infrared and NMR spectroscopy as soon as enough sample could be collected. The results are given below.

$Mn(CO)_5^-$. The infrared spectrum of the THF solution contained a number of closely spaced and overlapping bands in the CO stretching region. Bands were observed at approximately 2120, 2100, 2060, 2015, 1990, 1980 and 1930 cm^{-1}. The NMR spectrum consisted of a single resonance at δ —7.5 ppm. Evaporation of the solvent gave a solid yellow residue (0.78 g) whose infrared spectrum in CH_2Cl_2 had bands at 2075, 2030 and 1994 cm^{-1}. A comparison with the spectra of known samples and literature values for $HMn(CO)_5$ [11] and $Mn_2(CO)_{10}$

[12] indicates the solution contained these two materials in about an 8 : 1 molar ratio.

$CpFe(CO)_2^-$. The NMR spectra of the purple brown filtrate had a single resonance at δ 4.8 ppm and the infrared spectrum had bands at 1955, 1940 and 1756 cm^{-1} identical with the spectra of a known sample of $[CpFe(CO)_2]_2$. Evaporation of the solvent produced the dimer in nearly quantitative yields.

$Co(CO)_4^-$. The solution apparently contained a mixture of $Co_2(CO)_8$, $HCo(CO)_4$ and some salt of the $Co(CO)_4^-$ anion as judged by the infrared spectrum. Bands at 2115, 2070, 2055, 2045, 1890 and 1852 cm^{-1} were observed. On evaporation of the solvent and redissolving the residue in THF, the band at 2055 cm^{-1} disappeared and the band at 2115 cm^{-1} became much less intense while the intensity of the 1890 cm^{-1} band increased. A spectrum of a sample of $Co_2(CO)_8$ in THF run for comparison was very similar except that the intensity of the 1890 cm^{-1} band was considerably lower. Due to paramagnetic broadening no useful NMR spectra could be obtained. The solid residue obtained on evaporation of the THF weighed only 2.0 grams but the stripped solvent also gave a positive test for cobalt.

$CpMo(CO)_3^-$. The NMR spectrum of the solution contained resonances at δ 4.8 and —5.5 ppm in a 4.8 : 1 ratio and the infrared spectrum had bands at 2025 and 1928 cm^{-1} which were virtually identical to those of a known sample of $CpMo(CO)_3H$. A quantitative yield of the hydride was obtained upon evaporation of the solvent.

$CpW(CO)_3^-$. The results of this experiment were identical to those of the molybdenum analog. The NMR spectrum consisted of resonances at δ 4.9 and —7.2 ppm while two bands at 2021 and 1921 cm^{-1} were observed in the infrared. These were found to be identical to those observed for a pure sample of $CpW(CO)_3H$. A nearly quantitative yield of $CpW(CO)_3H$ was recovered.

Attempted oxidative addition reactions with formyl fluoride

$Rh(PPh_3)_3Cl$. To a suspension of 1.85 g (2.0 mmol) $Rh(PPh_3)_3Cl$ in 25 ml benzene at room temperature was added 0.1 g (2.1 mmol) HCOF. The suspension gradually turned deep yellow and after 1 h the yellow suspension was collected on a filter and washed with pentane, yielding 1.1 g $Rh(CO)(PPh_3)_2Cl$. A small amount of additional product was obtained by adding the pentane washes to the benzene filtrate. Upon evaporation of the pentane solutions 0.44 g triphenylphosphine was recovered.

$Ir(CO)(PPh_3)_2Cl$. In the same manner as described above 0.1 g (2.1 mmol) HCOF was added to a suspension of 1.56 g (2.0 mmol) $Ir(CO)(PPh_3)_2Cl$ in 25 ml benzene. The yellow suspension rapidly turned white, and after stirring for 20 minutes the suspended solid was collected on a filter, washed with pentane and dried. 1.6 g of $IrH(CO)(PPh_3)_2ClF$ was obtained.

$Ru(NO)(PPh_3)_2Cl$. A suspension of 2.07 g (3.0 mmol) $Ru(NO)(PPh_3)_3Cl$ in 25 ml benzene was treated with 0.15 g (3.1 mmol) HCOF. The green suspension immediately turned brown and after stirring an additional 30 min was filtered. The orange-brown solid recovered was washed several times with pentane and dried. The yield of $Ru(NO)(CO)(PPh_3)_2Cl$ was 1.5 grams. An additional 0.6 grams was obtained by the addition of pentane to the benzene filtrate. The crude product which was contaminated by a small amount of material with infra-

red absorptions at 1874 and 1583 cm^{-1} was purified by recrystallization from a benzene/hexane mixture.

Pt(PPh$_3$)$_4$. To a suspension of 2.49 g (2.0 mmol) of $Pt(PPh_3)_4$ in 25 ml dry benzene was added 0.1 g (2.1 mmol) HCOF. The suspension immediately went into solution and after stirring several minutes white crystals slowly began to form. The crystals (0.1 g) were collected on a glass frit and dried. 1.3 g of additional identical material was obtained by the addition of 100 ml pentane to the filtrate. The pentane filtrate was evaporated to dryness, yielding 1.1 g of pale yellow residue identified as triphenylphosphine.

Pd(PPh$_3$)$_4$. In the same manner as described above 2.31 g (2.0 mmol) $Pd(PPh_3)_4$ was treated with 0.1 g (2.1 mmol) HCOF. Upon addition of 100 ml pentane, 1.2 g of white microcrystalline product was obtained. 1.2 g of triphenylphosphine was recovered from the filtrate and wash solutions.

Ir(PMe$_3$)$_2$(CO)Cl. 0.1 g (2 mmol) HCOF was added to a solution of 0.81 g (2.0 mmol) $Ir(PMe_3)_2(CO)Cl$ in 25 ml benzene. The yellow color immediately faded and after 20 min the solution was filtered to remove a small amount of insoluble solids and the filtrate was evaporated to dryness. The solid residue was recrystallized from benzene/hexane, yielding 0.61 g of white crystalline product.

Attempted oxidative addition reactions of acetic formic anhydride

Rh(PPh$_3$)$_3$Cl. A solution of 0.18 g (2.0 mmol) $HCOOCOCH_3$ in 10 ml benzene was added dropwise to a suspension of 1.85 g (2.0 mmol) $Rh(PPh_3)_2Cl$ in 25 ml benzene at room temperature. After stirring for two hours the purple suspension gradually turned bright yellow and was collected on a filter. The solid was washed with pentane and dried yielding 1.0 grams. This material was confirmed as $Rh(CO)(PPh_3)_2Cl$ by comparison of its infrared spectrum with that of a known sample.

Ru(NO)(PPh$_3$)$_2$Cl. In the same manner as described above 1.38 g (2.0 mmol) $Ru(NO)(PPh_3)_2Cl$ was treated with 0.18 g (2 mmol) $HCOOCOCH_3$. The green suspension gradually turned orange brown over a period of an hour and was then filtered and washed with pentane. The yield of solid orange brown product was 1.1 g, Anal. Calcd. for $C_{37}H_{30}O_2NP_2ClRu$; C, 61.79; H, 4.20; N, 1.95; P, 8.61; Cl, 4.93; Ru, 14.05. Found: C, 60.90; H, 4.20; N, 1.85; P, 8.49; Cl, 5.33; Ru, 13.79%.

Pt(PPh$_3$)$_4$. To a suspension of 1.24 g (1 mmol) $Pt(PPh_3)_4$ in 20 ml benzene was added 0.1 g (1.1 mmol) $HCOOCOCH_3$ and the mixture stirred for 3 hrs. No apparent reaction took place and the $Pt(PPh_3)_4$ was recovered unchanged by evaporation of the solvent.

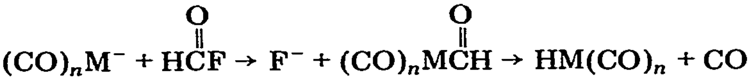
Pd(PPh$_3$)$_4$. Identical results were observed when $Pd(PPh_3)_4$ was substituted for the platinum complex.

Determination of the rate of decomposition of formyl fluoride

A solution containing approximately 0.5% formyl fluoride in THF was prepared at —10°C. The infrared spectrum of the solution was recorded from 2000—1600 cm^{-1} at intervals of 15 min over a two hour period. By comparison of the absorption of the solution at 1800 cm^{-1} the amount of decomposition over the two hour period was less than 5%. This experiment was repeated at room temperature and less than 10% decomposition was observed at one hour.

Results and discussion

The reaction of any of several different metal carbonyl anions with formyl fluoride did not result in the formation of any isolable neutral metal formyl complexes. Although the formation of NaF took place rapidly and smoothly the only other products identified were the corresponding metal carbonyl hydrides or metal carbonyl dimers. These results are similar to those reported by Casey and Newmann [13] and by Fiato and coworkers [14] using acetic formic anhydride. Based on this earlier work it is most likely, although by no means certain, that an unstable intermediate formyl complex is initially formed which rapidly decomposes to the metal carbonyl hydride.



$$(CO)_nM^- + H\overset{\overset{O}{\|}}{C}F \rightarrow F^- + (CO)_nM\overset{\overset{O}{\|}}{C}H \rightarrow HM(CO)_n + CO \qquad (1)$$

In the cases where the carbonyl hydrides are unstable further reaction takes place with the formation of hydrogen and the carbonyl dimers. We have shown that the decomposition of formyl fluoride to CO and HF is very slight under the conditions that the reactions were run making it unlikely that the hydrides are formed by the direct reaction of HF with the carbonyl anions.

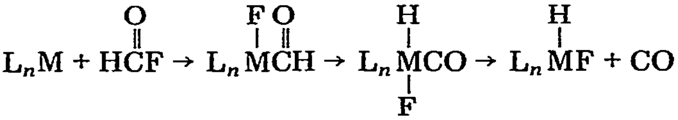
The oxidative addition reaction of formyl fluoride to unsaturated transition metal complexes also does not appear to be a general method of preparation of neutral formyl complexes. In most of the cases which were investigated, with two notable exceptions, the products obtained were the hydridofluorocomplexes, the same as those one might expect from the oxidative addition of HF to the metal complexes (Table 1). These reactions are quite rapid, generally going to completion in less than 10 minutes with a noticeable evolution of CO.

TABLE 1

PRODUCTS FROM FORMYL FLUORIDE REACTIONS

Starting complex	Reaction products	Selected IR data, mulls (cm^{-1})	Elemental analysis of metal complexes Found (calcd.) (%)
$Pt(PPh_3)_4$	$Pt(H)(F)(PPh_3)_2 + PPh_3 + CO$	2240w	C, 58.63 (58.45); H, 4.56 (4.22); P, 7.80 (8.37)
$Pd(PPh_3)_4$	$Pd(H)F(PPh_3)_2 + PPh_3 + CO$	—	C, 66.23 (66.42); H, 5.09 (4.82); P, 9.80 (9.57)
$Ir(CO)Cl(PMe_3)_2$	$IrH(CO)F(PMe_3)_2Cl + CO$	2172w, 2020s	C, 20.09 (19.65); H, 4.48 (4.39); P, 14.20 (14.48); Cl, 8.55 (8.29)
$Ir(CO)Cl(PPh_3)_2$	$IrH(CO)F(PPh_3)Cl + CO$	2240m, 2025s	C, 54.92 (55.53); H, 3.83 (3.90); P 7.80 (7.74); Cl; 4.38 (4.43)
$Ru(NO)Cl(PPh_3)_2$	$Ru(NO)(CO)Cl(PPh_3)_2 + HF$	1925s, 1593s	C, 61.86 (61.79); H, 4.51 (4.20); N, 1.95 (1.95); P, 8.80 (8.61); Cl, 5.33 (4.93)
$Rh(PPh_3)_3Cl$	$Rh(CO)Cl(PPh_3)_2 + PPh_3 + HF$	1980s	C, 64.34 (64.32); H, 4.10 (4.38); P 9.30 (8.97); Cl, 5.30 (5.13)

The decomposition of formyl fluoride under these conditions is much slower and cannot account for the formation of the HF adducts. The details of the mechanism of these reactions are not known and may be quite complex and different for each of the complexes. It seems quite likely, however, that a formyl fluoride complex is formed at some stage of the reaction which subsequently eliminates CO with the formation of the HF adduct.



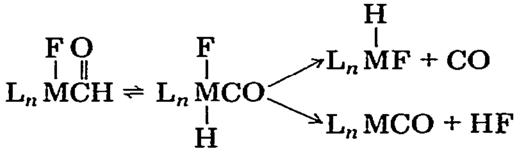
$$L_nM + H\overset{\overset{O}{\|}}{C}F \rightarrow L_n\overset{\overset{F}{|}}{M}\overset{\overset{O}{\|}}{C}H \rightarrow L_n\underset{\underset{F}{|}}{\overset{\overset{H}{|}}{M}}CO \rightarrow L_n\overset{\overset{H}{|}}{M}F + CO \tag{2}$$

The possible alternative, cleavage of the C—H bond, seems less likely under these mild conditions.

Based primarily on elemental analysis, the major product of the reaction between $Pt(PPh_3)_4$ and formyl fluoride, besides CO and PPh_3, is $HPtF(PPh_3)_2$. This product, which is difficult to obtain free of excess triphenyl phosphine, is a high melting (m.p. >230° C), nearly colorless solid which shows a rather weak infrared band at approximately 2240 cm^{-1} attributed to a Pt—H stretch. The properties are quite similar to those described for *trans*-$[PtHCl(PPh_3)_2]$ [15], which has been shown to be the product resulting from the reaction of HCl with $Pt(PPh_3)_3$ [16]. $Pt(PPh_3)_4$ readily dissociates a molecule of triphenyl phosphine to form $Pt(PPh_3)_3$ which can then react with formyl fluoride in a series of steps to form a five-coordinate $PtF(\overset{\overset{O}{\|}}{C}H)(PPh_3)_3$ complex. This intermediate upon dissociation of a second molecule of triphenyl phosphine and elimination of carbon monoxide (not necessarily in that order) would then yield *trans*-$PtH(F)(PPh_3)_2$. The reaction of $Pd(PPh_3)_4$ with formyl fluoride appeared to be exactly analogous to the platinum complex, although no well defined Pd—H stretching band could be observed in the infrared.

Both $Ir(CO)Cl(PPh_3)_2$ and $Ir(CO)Cl(PMe_3)_2$ give the six-coordinate HF adducts on reaction with HCOF. The complex formed with $Ir(CO)Cl(PPh_3)_2$ seems to be identical in every respect to the $HFIrCl(CO)(PPh_3)_2$ complex which Vaska obtained from the reaction of HF with $Ir(CO)Cl(PPh_3)_2$ [17]. The complex formed from $Ir(CO)Cl(PMe_3)_2$ displays infrared bands at 2020 and 2172 cm^{-1} which we have tentatively assigned to the CO and IrH stretching modes. The exact molecular configuration of this complex has not been determined; however, a structure with *trans* phosphines and a *cis* arrangement of the hydride and fluoride seems likely.

$Ru(NO)Cl(PPh_3)_2$ which is isoelectronic with $Ir(CO)Cl(PPh_3)_2$ also reacts readily with HCOF, giving a brown product. The infrared spectrum of this material shows very strong absorptions at 1925 and 1593 cm^{-1} which can be assigned to the CO and NO stretches of $Ru(NO)(CO)Cl(PPh_3)_2$. A comparison with an authentic sample [18] of the nitrosyl carbonyl derivative prepared independently confirmed the identity of the product. The product was contaminated by a very small amount of another material which exhibited infrared bands at 1874 and 1583 cm^{-1}, and gave a positive test for fluorine. This compound may be the HF adduct; however, it could not be isolated in the pure state for positive identification. The difference in behavior of the Ir and Ru complexes is interesting but not well understood. $Ru(NO)Cl(PPh_3)_2$ will oxidatively add HCl [19] and therefore presumably HF also, as will $Ir(CO)Cl(PPh_3)_2$ [17]. If the reaction proceeds through a fluoroformyl intermediate, a logical following step would be the formation of a CO—H—F complex (eq. 3).



$$L_n\overset{\mathrm{F}}{\overset{|}{\mathrm{M}}}\overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}}\mathrm{H} \rightleftharpoons L_n\underset{\underset{\mathrm{H}}{|}}{\overset{\overset{\mathrm{F}}{|}}{\mathrm{M}}}\mathrm{CO} \begin{cases} \rightarrow L_n\overset{\overset{\mathrm{H}}{|}}{\mathrm{M}}\mathrm{F} + \mathrm{CO} \\ \rightarrow L_n\mathrm{MCO} + \mathrm{HF} \end{cases} \qquad (3)$$

This would then either dissociate a CO ligand or reductively eliminate HF, the former pathway being the preferred one for the Ru complex but the latter being favored for the Ir compound, even though both are capable of forming stable five-coordinate CO adducts or six-coordinate HF addition products.

$Rh(PPh_3)_3Cl$ also forms a carbonyl complex upon reaction with formyl fluoride. The yellow compound which is formed quantitatively contains a CO stretching band in the infrared at 1980 cm^{-1}, and has been positively identified as $Rh(CO)(PPh_3)_2Cl$. Along with this an equivalent of triphenylphosphine can also be recovered. A plausible intermediate in this case would be $Rh(CO)H(PPh_3)_2$·ClF. The very similar complex $Rh(CO)H(PPh_3)_2Cl_2$ has been shown to be very unstable and decomposes readily to $Rh(CO)(PPh_3)_2Cl$ and HCl [20] in the same manner which we have proposed. It is interesting to note that there are no further reactions of the $Rh(CO)(PPh_3)_2Cl$ with any excess formyl fluoride which is present even though the analogous iridium complex reacts quite readily.

Because of the obvious similarities of formyl fluoride with acetic formic anhydride we thought a comparison between the mode of reaction of these two compounds with some of the transition metal complexes would be informative. The reaction of acetic formic anhydride with $Ir(CO)Cl(PPh_3)_2$ had already been investigated and was found to give a number of products depending on conditions [21]. When one equivalent of $HCOOCOCH_3$ is used, an equimolar mixture of $Ir(CO)Cl_2(PPh_3)_2H$ and $Ir(CO)_2(PPh_3)_2H$ along with a mole of acetic anhydride is formed. A key step proposed in this reaction is the formation of an unstable formyl acetate complex which is similar to the formyl fluoride complex which we have postulated.

Although neither $Pt(PPh_3)_4$ or $Pd(PPh_3)_4$ readily react with acetic formic anhydride both $Rh(PPh_3)_3Cl$ and $Ru(PPh_3)_2(NO)Cl$ are rapidly converted to the CO derivatives $Rh(CO)(PPh_3)_2Cl$ and $Ru(NO)(CO)(PPh_3)_2Cl$, respectively. The formation of carbonyl-metal complexes from formyl fluoride and acetic formic anhydride is reminiscent of the decarbonylation of aldehydes, a reaction in which both rhodium [6,7] and ruthenium [22] complexes are effective reagents. Although the aldehyde decarbonylation reaction takes place through the oxidative addition across the aldehyde C—H bond there is not sufficient evidence to determine the mode of addition of either formyl fluoride or acetic formic anhydride.

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