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Tetraethylammonium fluoride as a convenient source of hydroxide ions in acetonitrile solution. Its reaction with $[Fe(\eta-C_5H_5)(CO)_2(L)]^+$ salts and $[Fe(\eta-C_5H_5)(CO)_2X]$ complexes

Stephen Byrne, M. Gary Cox, Anthony R. Manning *

Department of Chemistry, University College Dublin, Belfield, Dublin 4, Ireland

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Abstract

In acetonitrile solution, $[Et_4N]F{\cdot}2.5H_2O$ acts as a convenient source of hydroxide ions for use in organometallic chemistry. Within 20 min, at room temperature, it converts $[Fe(\eta-C_5H_5)(CO)_2(L)]^+$ salts (molar ratio 1:1) to $[Fe_2(\eta-C_5H_5)_2(CO)_3(L)]$ (L = CO, CNMe and PPh_3) complexes in good yields with the evolution of CO_2 and, when L = CO, CO. Under the same conditions it converts $[Fe(\eta-C_5H_5)(CO)_2Cl]$ (molar ratio 1:2 or, better, 1:3) to $[Fe_2(\eta-C_5H_5)_2(CO)_3(NCMe)]$. Other $[Fe(\eta-C_5H_5)(CO)_2X]$ (X = Br or I) react only slowly to give $[Fe_2(\eta-C_5H_5)_2(CO)_4]$. Other routes to $[Fe_2(\eta-C_5H_5)_2(CO)_3(PPh_3)]$ and $[Fe_2(\eta-C_5H_5)_2(CO)_3(NCMe)]$ involve prolonged photolysis and give impure or non-isolable products. This route gives them as analytically pure solids in high yields. It is proposed that in acetonitrile solutions, the F^- ions react with H_2O to give HF and OH^- in a reaction driven by the strength of the H–F bond and the lack of anion solvation in acetonitrile. The OH^- ions then attack the $[Fe(\eta-C_5H_5)(CO)_2(L)]^+$ cations at CO with the evolution of CO_2 to give the $[Fe(\eta-C_5H_5)(CO)(L)]^-$ which subsequently displaces L from the cations. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

In a previous publication from these laboratories [1], it was shown that commercial $[Et_4N]F{\cdot}nH_2O$ ($n = 2$–3) in tetrahydrofuran or dichloromethane solutions acted as a source of F^- ions, but in acetonitrile it acted as a source of $[OH]^-$. Thus, with $[Fe_2\{\eta,\eta-C_5H_4CH_2CH(NMe_3)C_5H_4\}(CO)_2(\mu-CO)_2][SO_3CF_3]$ in acetonitrile it gives $[Fe_2\{\eta,\eta-C_5H_4CH_2CH(NMe_3)C_5H_4\}(CO)_2(\mu-CO)_2][OH]$; with R,S'-$[Fe_2\{\eta,\eta-C_5H_4CH(NMe_2)CH(NMe_3)C_5H_4\}(CO)_2(\mu-CO)_2][SO_3CF_3]$ in tetrahydrofuran it gives R,S'-$[Fe_2\{\eta,\eta-C_5H_4CH(NMe_2)CH(NMe_3)C_5H_4\}(CO)_2(\mu-CO)_2][H_4F_5]$, but in acetonitrile it brings about a Hoffman elimination to give the enamine $[Fe_2\{\eta,\eta-C_5H_4CHCH(NMe_2)C_5H_4\}(CO)_2(\mu-CO)_2]$ and NMe_3; and with $R,R'/S,S'$-$[Fe_2\{\eta,\eta-C_5H_4CH(NMe_2)CH(NMe_3)C_5H_4\}(CO)_2(\mu-CO)_2][SO_3CF_3]$ it gives $R,R'/S,S'$-$[Fe_2\{\eta,\eta-C_5H_4CH(NMe_2)CH(NMe_3)C_5H_4\}(CO)_2(\mu-CO)_2][H_5F_6]$ in dichloromethane, but in acetonitrile $[Fe_2\{\eta,\eta-C_5H_4CH_2CH(NMe_2)C_5H_4\}(CO)_2(\mu-CO)_2]$ is formed in a Water–Gas-Shift reaction.

These observations prompted us to investigate the use of $[Et_4N]F{\cdot}nH_2O$ in acetonitrile solution as a convenient and clean source of $[OH]^-$ ions in organometallic chemistry. We have looked at its reactions with various metal–carbonyl complexes and report here on those with $[Fe(\eta-C_5H_5)(CO)_2(L)]^+$ salts (L = CO, PPh_3 and CNMe) and $[Fe(\eta-C_5H_5)(CO)_2X]$ complexes (X = Cl, Br and I).

2. Experimental

Published methods or extensions thereof were used to prepare $[Fe(\eta-C_5H_5)(CO)_3][BF_4]$ [2], $[Fe(\eta-C_5H_5)(CO)_2$-

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* Corresponding author. Tel.: + 353-1-7062311; fax: + 353-1-7062127.

E-mail address: anthony.manning@ucd.ie (A.R. Manning).

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$(PPh_3)]Cl\cdot 3H_2O$ [3], $[Fe(\eta\text{-}C_5H_5)(CO)_2(CNMe)][SO_3CF_3]$ [4], $[Fe(\eta\text{-}C_5H_5)(CO)_2Cl]$ [5], $[Fe(\eta\text{-}C_5H_5)(CO)_2Br]$ [5,6] and $[Fe(\eta\text{-}C_5H_5)(CO)_2I]$ [7–9]. Other chemicals were purchased and used as received.

All reactions were carried out at room temperature (r.t.) under an atmosphere of nitrogen and monitored by IR spectroscopy.

2.1. The reactions of $[Fe(\eta\text{-}C_5H_5)(CO)_2(L)]X$ salts and $[Et_4N]F\cdot 2.5H_2O$

Solid $[Et_4N]F\cdot 2.5H_2O$, (0.33 g, 1.7 mmol) or its solution in MeCN (15 cm^3) was added to a solution of $[Fe(\eta\text{-}C_5H_5)(CO)_3][BF_4]$, (0.5 g, 1.7 mmol) in MeCN (80 cm^3). A rapid reaction took place with the evolution of CO and CO_2, which was detected by IR spectroscopy. The solution changed colour from yellow to red. The reaction was complete within 5 min to give $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_2(\mu\text{-}CO)_2]$ as the sole product. It was isolated in > 95% yield from the filtered reaction mixture by removal of the solvent at reduced pressure and recrystallization of the residue from toluene–hexane mixtures. If an excess of $[Et_4N]F\cdot 2.5H_2O$ was used, the reaction was faster, but the product remained the same. No reaction took place if the reaction was carried out in dry THF or in the absence of H_2O, e.g. using anhydrous KF/18-crown-6 in rigorously dried MeCN. $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_2(\mu\text{-}CO)_2]$ was identified by elemental analysis and spectroscopy [7,11]. When the final reaction mixtures were washed with H_2O, the aqueous layers were acidic to litmus paper.

A solution of $[Et_4N]F\cdot 2.5H_2O$ (0.29 g, 1.24 mmol) in MeCN was added to one of $[Fe(\eta\text{-}C_5H_5)(CO)_2(PPh_3)]Cl\cdot 3H_2O$ (0.657 g, 1.24 mmol) also in MeCN (80 cm^3). CO_2 was evolved and the solution changed colour from yellow to green. The reaction was complete within 20 min to give $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(PPh_3)]$ as the major product together with traces of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_4]$. Partial removal of the solvent at reduced pressure followed by storage at − 10°C gave $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(PPh_3)]$ as a green solid that was filtered off and recrystallized from MeCN (yield 65%). The original filtrate was concentrated at reduced pressure to give $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_4]$ (yield 14%), much of which appeared to be formed during the work-up. $[Fe_2(\eta\text{-}C_5H_5)_2(CO)(PPh_3)(\mu\text{-}CO)_2]$ (m.p. 186–189°C) was identified by elemental analysis. Anal. Found: C, 62.8; H, 4.3; P, 5.8. Calc. for $[Fe_2(\eta\text{-}C_5H_5)_2(CO)(PPh_3)(\mu\text{-}CO)_2]$: C, 63.2; H, 4.2; P, 5.3%. IR (MeCN, cm^{-1}): ν(C=O) 1948 (8.7), 1727 (10) with relative peak heights in parentheses. ^{1}H-NMR(δ ppm): cyclopentadienyl protons at 4.16 (5), 4.19 (5) at 313 K, and 3.82 (1.6), 4.05 (3.4), 4.31 (5) at 243 K with integrations in parentheses; coalescence temperature 282 K (toluene-D_8 solution). It has been obtained previously in an impure state [12].

Using the same procedure, $[Fe(\eta\text{-}C_5H_5)(CO)_2(CNMe)][BF_4]$ was completely converted to $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(CNMe)]$ [13] as the sole product within 5 min and CO_2 was evolved. The former was isolated and purified in the same way as $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_4]$ and identified by analysis and spectroscopy (yield > 95%).

$[Fe(\eta\text{-}C_5H_5)(CO)(Ph_2PCH_2CH_2PPh_2)]Cl$ failed to react with $[Et_4N]F\cdot 2.5H_2O$ in MeCN.

2.2. The reaction of $[Et_4N]F\cdot 2.5H_2O$ with $[Fe(\eta\text{-}C_5H_5)(CO)_2\ X]$ (X = Cl, Br and I)

The addition of a solution of $[Et_4N]F\cdot 2.5H_2O$ (1.09 g, 4.70 mmol) in MeCN (15 cm^3) to one of $[Fe(\eta\text{-}C_5H_5)(CO)_2Cl]$ (0.5 g, 2.35 mmol) in MeCN (50 cm^3) (molar ratio 2:1) gave a mixture of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_4]$ and $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(NCMe)]$. The reaction was complete within 10 min, but was nearly instantaneous when a three-fold excess of $[Et_4N]F\cdot 2.5H_2O$ was used. $[Fe_2(\eta\text{-}C_5H_5)_2(CO)(NCMe)(\mu\text{-}CO)_2]$ was then the sole product and it could be isolated as an analytically pure green solid in > 60% yield by immediate removal of the solvent at reduced pressure followed by washing of the residue with cold EtOH. If the work-up was delayed, large amounts of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_4]$ were also formed and the two were impossible to separate. When this reaction was carried out in PhCN or $PhCH_2CN$ solutions, it was much slower even with a large excess of $[Et_4N]F\cdot 2.5H_2O$. Consequently, the $[Fe_2(\eta\text{-}C_5H_5)_2(CO)(NCR)(\mu\text{-}CO)_2]$ was contaminated with varying amounts of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_2(\mu\text{-}CO)_2]$ which could not be separated from it. $[Fe_2(\eta\text{-}C_5H_5)_2(CO)(NCMe)(\mu\text{-}CO)_2]$ was identified by elemental analysis. Anal. Found: C, 49.0; H, 3.3; N, 3.2. Calc. for $[Fe_2(\eta\text{-}C_5H_5)_2(CO)(NCMe)(\mu\text{-}CO)_2]$: C, 48.2; H, 3.5; N, 3.8%. IR (MeCN, cm^{-1}): (νC=O) 1941 (6.7), 1742 (10) with relative peak heights in parentheses. Its low stability prevented the measurement of its NMR spectrum. This compound has been prepared previously but was not isolated or characterized in any way [14].

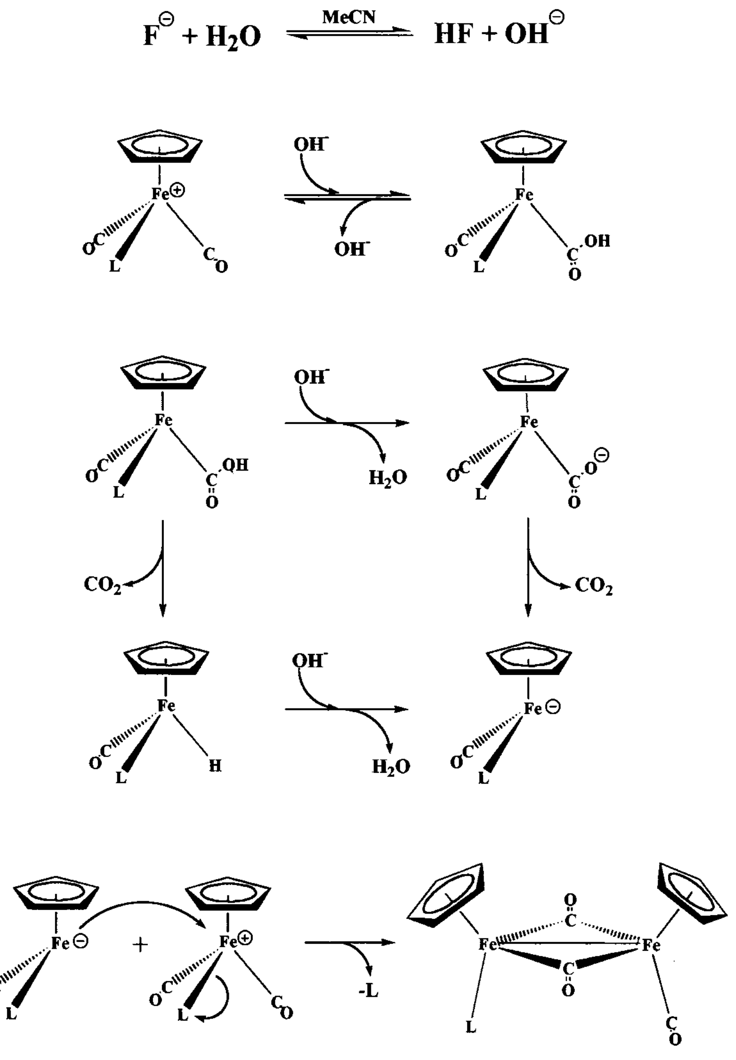
If $[Fe(\eta\text{-}C_5H_5)(CO)_2Cl]$ was replaced by $[Fe(\eta\text{-}C_5H_5)(CO)_2Br]$ or $[Fe(\eta\text{-}C_5H_5)(CO)_2I]$, the reactions were very much slower and the sole product was $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_2(\mu\text{-}CO)_2]$ even when a large excess of $[Et_4N]F\cdot 2.5H_2O$ was used. The rate of reaction declined along the series $[Fe(\eta\text{-}C_5H_5)(CO)_2Cl] > [Fe(\eta\text{-}C_5H_5)(CO)_2Br] > [Fe(\eta\text{-}C_5H_5)(CO)_2I]$.

3. Results and discussion

Commercial tetraalkylammonium fluorides are hydrates of somewhat indeterminate composition [15]. We have used both $[Me_4N]F\cdot xH_2O$ and $[Et_4N]F\cdot yH_2O$ in the course of this work, but the former is deliquescent and so we preferred the latter. Its composition was determined by elemental analysis and our sample was shown to be $[Et_4N]F \cdot 2.5H_2O$.

The reaction of $[Fe(\eta\text{-}C_5H_5)(CO)_3][BF_4]$ with an equimolar amount of $[Et_4N]F \cdot 2.5H_2O$ in acetonitrile, but not tetrahydrofuran gives $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_2(\mu\text{-}CO)_2]$ as the sole product in $> 95\%$ isolated yields whilst IR studies suggest that the reaction is quantitative and that CO and CO_2 are evolved in comparable amounts. $[Fe(\eta\text{-}C_5H_5)(CO)_2(CNMe)][SO_3CF_3]$ gives $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(CNMe)]$ quantitatively, and $[Fe(\eta\text{-}C_5H_5)(CO)_2(PPh_3)]Cl \cdot 3H_2O$ gives $[Fe_2(\eta\text{-}C_5H_5)_2(CO)(PPh_3)(\mu\text{-}CO)_2]$. In both cases, CO_2 but not CO is evolved. The rate of the reaction decreases along the series $L = CO > CNMe \sim PPh_3$ whilst $[Fe(\eta\text{-}C_5H_5)(CO)(Ph_2PCH_2CH_2PPh_2)]Cl$ fails to react.

The formation of CO_2 suggests that the most plausible pathway for this reaction is that shown in Scheme 1. It is similar to that proposed to account for the formation of $[Mn_2(\eta^6\text{-}C_6Me_6)_2(CO)_2(\mu\text{-}CO)_2]$ from $[Mn(\eta^6\text{-}C_6Me_6)(CO)_3]^+$ and $[BH_4]^-$ in the presence of water [18], and of $[Fe_2(\eta^5\text{-}C_6H_7)_2(CO)_2(\mu\text{-}CO)_2]$ from $[Fe(\eta^5\text{-}C_6H_7)(CO)_3]^+$ and a large excess of $[Me_4N]OH \cdot 5H_2O$ [18]. In all three reactions, the reactive species are hydroxide ions. In the present case, they are formed in acetonitrile by the reaction of F^- ions with H_2O. This reaction is driven by the formation of the very strong H–F bond coupled with limited anion solvation in acetonitrile. Alternatively, it may be more accurate to represent this step as a polarization of the water molecule by F^- rather than complete ionization. It is consistent with the proposed scheme that the reactions do not take place in solvents such as tetrahydrofuran



Scheme 1.

which solvate anions more effectively or in the absence of water, e.g. with anhydrous KF/18-crown-6 in dry acetonitrile. HF is also formed and is responsible for the acidity observed in the aqueous layer when the reaction mixtures are washed with water.

The attack by $[OH]^-$ ions on coordinated CO is well documented. $[Fe(\eta\text{-}C_5H_5)(CO)_2L]^+$ salts (L = CO or PPh_3) have been reported to react with KOH to give isolable $[Fe(\eta\text{-}C_5H_5)(CO)(L)(CO_2H)]$ and $K[Fe(\eta\text{-}C_5H_5)(CO)(PPh_3)(CO_2)]$ [16,17]. The subsequent decarboxylation may proceed via $[Fe(\eta\text{-}C_5H_5)(CO)(L)(CO_2H)]$ or $[Fe(\eta\text{-}C_5H_5)(CO)(L)(CO_2)]^-$ to $[Fe(\eta\text{-}C_5H_5)(CO)(L)H]$ or $[Fe(\eta\text{-}C_5H_5)(CO)(L)]^-$, respectively.

The formation of CO in comparable amounts to CO_2 when L = CO and its absence when L = CNMe or PPh_3 is the evidence for nucleophilic attack by $[Fe(\eta\text{-}C_5H_5)(CO)(L)]^-$ on $[Fe(\eta\text{-}C_5H_5)(CO)_2(L)]^+$ with displacement of the weakest π-acceptor ligand, L, from the cation. $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_4]$ may be a primary product from the reaction when L = PPh_3, but we think that it arises from the decomposition of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)(PPh_3)(\mu\text{-}CO)_2]$ as its concentration in the reaction mixture increases with time.

The proposed scheme rationalizes satisfactorily the ligand dependence of the reaction rate which, for the $[Fe(\eta\text{-}C_5H_5)(CO)_2(L)]^+$ cations, decreases in the order L = CO > CNMe and PPh_3. This dependency may be linked to the frequencies of the $\nu(C{=}O)$ vibrations of the cations which decrease 2074/2124 cm^{-1} (L = CO), 2019/2064 cm^{-1} (L = CNMe), and 2014/2057 cm^{-1} (L = PPh_3). This is not surprising as the $\nu(C{=}O)$ frequencies and the electrophilicities of the CO groups decrease with increasing back-bonding into the CO π*-orbitals as L changes from CO to CNMe or PPh_3. It also explains why there is no reaction between $[Fe(\eta\text{-}C_5H_5)(CO)(Ph_2PCH_2CH_2PPh_2)]Cl$ and $[Et_4N]F\cdot 2.5H_2O$ as the metal centre is electron rich, which discourages nucleophilic attack at the carbonyl C atom.

Titration experiments have shown that the stoichiometry of these reactions is 1:1. However, all are faster if a three-fold excess of $[Et_4N]F\cdot 2.5H_2O$ is used. This is to be expected as the concentration of $[OH]^-$ ions would be much higher and would facilitate relevant steps in the scheme, particularly when L is the stronger donor, PPh_3, and towards the end of the reaction.

The same scheme with $L = X^-$ may be used to account for the products from the reactions of $[Fe(\eta\text{-}C_5H_5)(CO)_2X]$ provided that $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(X)]^-$, solvolyses to $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(NCMe)]$ (X = Cl) or decomposes to $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_4]$ (X = Br or I). However, this does not account for the markedly different reaction rates for $[Fe(\eta\text{-}C_5H_5)(CO)_2Cl]$ as compared with its bromo or iodo counterparts. The $\nu(C{=}O)$ frequencies of the three compounds {$[Fe(\eta\text{-}C_5H_5)(CO)_2Cl]$ 2015/2054 cm^{-1}; $[Fe(\eta\text{-}C_5H_5)(CO)_2Br]$ 2010/2052 cm^{-1}; $[Fe(\eta\text{-}C_5H_5)(CO)_2I]$ 2009/2047 cm^{-1}}suggest that the electrophilicities of the CO ligands are all quite low and do not differ greatly. Consequently, a second pathway must operate when X = Cl, though not when X = Br or I, and we suggest that in it the reactive species is $[Fe(\eta\text{-}C_5H_5)(CO)_2(NCMe)]^+$ (Scheme 1, L = NCMe). It is well-known that the Cl^- ligand in $[Fe(\eta\text{-}C_5H_5)(CO)_2Cl]$ is labile, e.g. unlike $[Fe(\eta\text{-}C_5H_5)(CO)_2X]$ (X = Br or I) it dissolves in water with the formation of $[Fe(\eta\text{-}C_5H_5)(CO)_2(OH_2)]^+Cl^-$, so it is not unreasonable to suggest that a similar solvolysis takes place in acetonitrile even if only to a very limited extent. $[Fe(\eta\text{-}C_5H_5)(CO)_2(NCMe)]^+$ would be expected to react much more rapidly than the covalent halo-complexes due to its more electrophilic CO groups as reflected in its higher $\nu(C{=}O)$ frequencies (2029/2074 cm^{-1}). However, this does not explain why two moles of $[Et_4N]F\cdot 2.5H_2O$ are required to bring about a complete reaction of $[Fe(\eta\text{-}C_5H_5)(CO)_2Cl]$.

3.1. NMR spectra of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(L)]$

The NMR spectra of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(L)]$ {L = CO [18], CNMe [11,19], $P(OPh)_3$ [20] and CN^- [12]} have been reported and discussed in detail, and have been instrumental in revealing the mechanism of fluxionality in metal–carbonyl compounds. However, comparable studies have not been carried out on $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(PPh_3)]$, perhaps because it was reported to be unstable [10]. However, we have found that when $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(PPh_3)]$ is pure, it is sufficiently stable in toluene-d_8 solution to allow its ^1H-NMR spectrum to be obtained at various temperatures. Unfortunately, only the cyclopentadienyl resonances could be studied. These are slightly broadened so that $^{31}P-^1H$ coupling was not detected, and a ^{13}C-NMR spectrum could not be obtained.

Spectroscopic data suggests that $[Fe_2(\eta\text{-}C_5H_5)_2(CO)(PPh_3)(\mu\text{-}CO)_2]$ has the same structure as that found for $[Fe_2(\eta\text{-}C_5H_5)_2(CO)\{P(OPh)_3\}(\mu\text{-}CO)_2]$ [21]. There are two species present in its toluene-d_8 solutions. At low temperatures, they are present in the ratio of 2.2:1 and each gives rise to a pair of singlet cyclopentadienyl resonances which are due to the C_5H_5 ligands bound to Fe(CO) and $Fe(PPh_3)$, respectively. The two resonances at higher• are coincident so that three peaks are observed. The two species are probably *cis*- and *trans*-isomers, but it is not possible to establish which is the major and which is the minor one. On warming, isomer exchange becomes faster, the cyclopentadienyl resonances broaden and coalesce so that when the fast exchange limit is reached at ca. 40°C, these protons give rise to two equal resonances, which are due to the two types of C_5H_5 groups. The coalescence temperature (9°C, 282 K) can be used to obtain the ΔG^* values for the fluxional *cis–trans* isomerism [22]. These are 15.7 kcal mol^{-1} from the major and 15.0 kcal mol^{-1} from the minor isomer. Although they are larger than the corresponding values for $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_4]$, (ca. 10.4 kcal mol^{-1} [18c]), these values are comparable to that of ca. 13.7 kcal mol^{-1} for $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3\text{-}\{P(OPh)_3\}]$ [20], and it is probale that the mechanism of the fluxional process which brings about the interchange of *cis*- and *trans*-isomers of $[Fe_2(\eta\text{-}C_5H_5)_2\text{-}(CO)_3(PPh_3)]$ is identical with that proposed for $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3\{P(OPh)_3\}]$ [20].

We were unable to obtain the ^{1}H-NMR spectrum of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(NCMe)]$.

4. Conclusions

In acetonitrile solution, $[Et_4N]F\cdot 2.5H_2O$ is a convenient source of OH^- ions, and is a useful reagent provided that the HF, which is also formed, does not affect the reaction.

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