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THE REACTIVITY OF CYCLOHEXADIENYL(CYCLOPENTADIENYL)IRON DERIVATIVES

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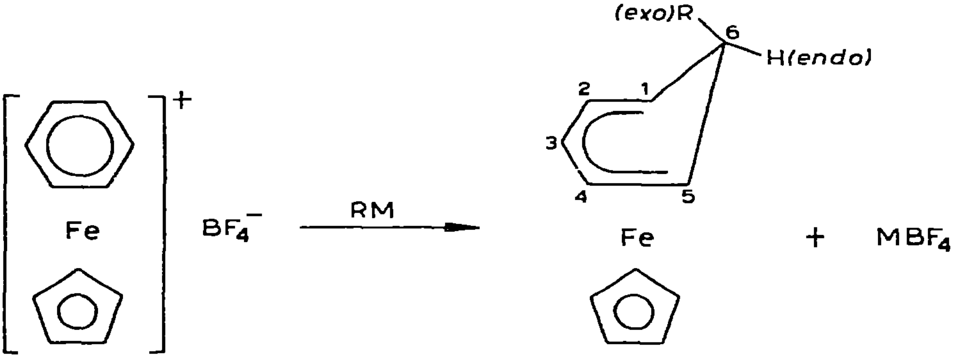
Summary

Oxidation of *exo*-substituted (cyclohexadienyl)cyclopentadienyliron derivatives, *exo*-$RC_6H_6FeC_5H_5$ ($R = C_2H_5$, $C_6H_5CH_2$ or C_5H_5), by $(Ph_3C)BF_4$ or bromosuccinimide proceeds by either of two routes, *exo*-R abstraction or *endo*-H abstraction. Mixtures of $[C_6H_6FeC_5H_5]^+$ and $[RC_6H_5FeC_5H_5]^+$ are formed in the reactions. The tendency for R-abstraction rises along the series $C_5H_5 < C_2H_5 < C_6H_5CH_2$. When heated, the compounds (arene H) FeC_5H_5 are transformed to ferrocene.

In the previous paper [1] we studied the reaction of benzene(cyclopentadienyl)iron tetrafluoroborate with a number of organometallic species RM (R = Na, Li, MgHal), which produces 6-substituted derivatives of cyclohexadienyl(cyclopentadienyl)iron (eqn. 1):



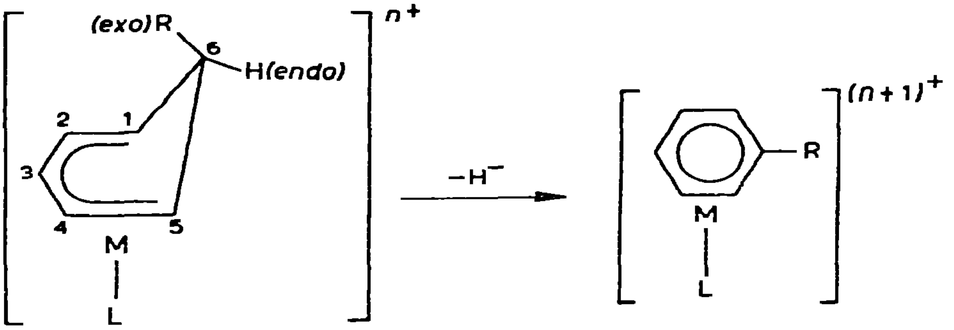
(1)

(Ia-Id)

(Ia, $R = CH_3$; Ib, $R = C_2H_5$; Ic, $R = C_6H_5CH_2$; Id, $R = C_5H_5$)

Below, reactions of the π-complexes Ia-Id with hydride ion acceptors are described.

Hydride ion abstraction is quite characteristic of cyclohexadienyl derivative of transition metals: the cyclohexadienyl ligand is transformed into the arene ligand and the oxidation state of the metal increases by one (eqn. 2).



(2)

This reaction has been described for various cyclohexadienyl π-complexes, e.g., L = $(CO)_3$, M = Re, Mn, R = H [2, 3]; L = tetraarylcyclobutadiene, M = Co, R = H, Alk [4]; L = C_5H_5, M = Fe, R = H [5-7], R = CH_3, C_6H_5 [8]; L = arene, M = Fe, R = tert-C_4H_9, C_6H_5, CH_2=CH— [9]*.

If, R = H, reaction 2 will give the unsubstituted arene derivative. If R = Alk or Ar, then usually a π-complex containing a substituent in the arene ligand will be formed.

Possible hydride ion acceptors are $(Ph_3C)BF_4$ [6-8], bromosuccinimide (BS) [4, 6-8], CCl_4 [2, 5, 10] and $AlCl_3$ [3]. The most widely-used are $(Ph_3C)BF_4$ and BS.

It is known [4, 8] that BS can abstract both the *exo*-hydrogen (R = H, eqn. 2) and the *endo*-hydrogen (R ≠ H). As for $(Ph_3C)BF_4$, this reactant was reported to be in some cases stereospecific, that is, inactive towards compounds containing only the *endo*-hydrogen (R ≠ H). For example, the following cyclohexadienyl π-complexes could not be converted into the respective arene derivatives by the action of $(Ph_3C)BF_4$: *exo*-$(CH_3)_nC_6H_{7-n}FeC_5H_5$, *exo*-$C_6H_5C_6(CH_3)_nH_{6-n}FeC_5H_5$ [8], $CH_3C_6H_6Mn(CO)_3$ [11].

No C—C bond fission at $C_{(6)}$ (eqn. 2) in the course of transformation of a cyclohexadienyl ligand into the arene ligand has been reported for R = Alk or Ar, although for R = *exo*-CN [12] or *exo*-$C(COOC_2H_5)_2$ [13] under the action of $(Ph_3C)BF_4$ this occurs.

However, fission of this kind is quite facile when the ligand is cyclopentadiene [14]. In this case, the *exo*-radical is abstracted while the *endo*-hydrogen is not (eqn. 3).

We have thoroughly studied the reaction of cyclohexadienyl(cyclopentadienyl)iron derivatives Ia-Id with $(Ph_3C)BF_4$ and BS. The reactions were carried out in a dry argon atmosphere in absolute solvents since special experiments

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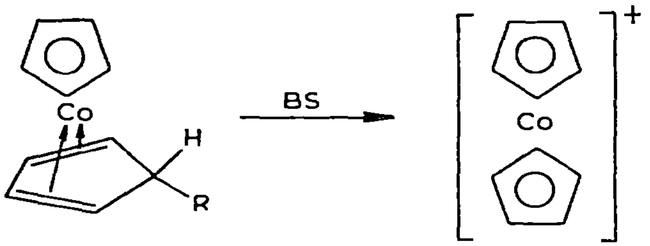
* In the last case, the H^- abstraction is accompanied by decomposition of the π-complex and formation of a substituted non-coordinated arene (the oxidant is $KMnO_4$).

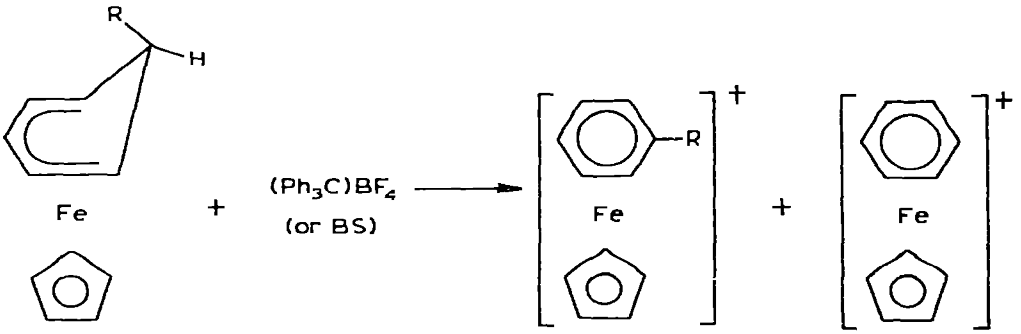


(3)

(R = CCl_3 , C_6H_5CO)

have shown that the action of O_2 in solution (i) transforms the cyclohexadienyl species into cations and then (ii) decomposes them entirely. Typical runs took 30 min at about 20°. The reaction of Ia-Id with $(Ph_3C)BF_4$ was performed in methylene chloride and the resulting cations were precipitated in the form of tetrafluoroborates. The BS reaction was carried out in a mixture of light petroleum with methanol containing $NaPF_6$ and the products were isolated as the hexafluorophosphates. As soon as the reaction was complete the reaction mixture was studied by thin-layer chromatography on Al_2O_3.

Two products are obtained when compounds Ib-Id are treated with BS or $(Ph_3C)BF_4$. The first, due to *endo*-6-hydrogen abstraction, is the substituted arene(cyclopentadienyl)iron cation (IIb-IId, eqn. 4). The other, due to abstraction of the *exo*-hydrocarbon radical from $C_{(6)}$ of the cyclohexadienyl compound, is the unsubstituted benzene(cyclopentadienyl)iron cation (III).



(4)

(I) (II) (III)

(b, R = C_2H_5; c, R = $C_6H_5CH_2$; d, R = C_5H_5)

(Ia, R = CH_3, gives only IIa, R = CH_3

Ib treated with either $(Ph_3C)BF_4$ or BS gives a mixture of IIb and III in the total yield of about 80%. The oxidation is accompanied with decomposition. The mixture of IIb and III tetrafluoroborates cannot be separated quantatively, on alumina, but the Al_2O_3 thin-layer chromatograms may be used to determine the composition of the reaction mixtures. Also, the mixture of tetrafluoroborates was pyrolysed and the resulting arenes were studied by GLC. No exact

IIb/III ratio could be found, but it was shown that a large excess of the oxidant raised the proportion of III in the mixture, made the decomposition more active, and lowered the total cation yield.

The cyclohexadienyl π-complex Ic when acted upon by BS is totally oxidised to the unsubstituted III, with the C—C bond being broken. The oxidation is accompanied by strong decomposition and the yield of III is below 50%. Oxidation using $(Ph_3C)BF_4$ also leads in general to III (yields are up to 68%), but in some cases an insignificant amount of IIc may be found. Both complexes were isolated by chromatography on Al_2O_3.

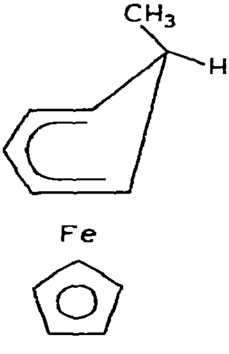
Under the action of $(Ph_3C)BF_4$ compound Id, containing a cyclopentadienyl substituent at $C_{(6)}$, forms a mixture of IId and III (2/1) in all cases, which was separated on Al_2O_3. The reaction with BS is more complex, and leads to a mixture of III with a substituted $[areneFeC_5H_5]^+$ compound. The second cation is not identical to the expected $[C_5H_5C_6H_5FeC_5H_5]^+$ (IIId); probably, it is a product of addition of an HBr molecule to IId, $[BrC_5H_6C_6H_5FeC_5H_5]^+$.

Finally, the *exo*-substituted cyclohexadienyl π-complex Ia acted upon by either $(Ph_3C)BF_4$ or BS gives almost entirely the substituted cation IIa. The C—C bond fission in this case, especially with $(Ph_3C)BF_4$, is quite insignificant.

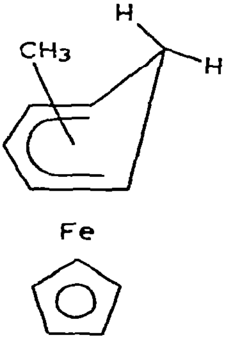
These results suggest that the tendency of Ia-Id to lose their hydrocarbon *exo*-substituents increases Ia $\leqslant$ Id < Ib < Ic.

All four compounds are able to loose their *endo*-hydrogens under the action of either $(Ph_3C)BF_4$ or BS. No stereospecificity could be observed with $(Ph_3C)BF$

Further, we have applied $(Ph_3C)BF_4$ to the oxidation of IV, an isomer of Ia, in which the CH_3 group is attached to one of the five coplanar $C_{(1-5)}$ atoms rather than to the methylene $C_{(6)}$ atom.



(Ia)



(IV)

The compound was obtained from $[CH_3C_6H_5FeC_5H_5]BF_4$ treated with $LiAlH_4$ in dimethoxyethane. (Previously [6] $NaBH_4$ was used.) The action of $(Ph_3C)BF_4$ on IV gave $[CH_3C_6H_5FeC_5H_5]^+$.

It seems that the interaction of *exo*-$RC_6H_6FeC_5H_5$ with oxidants strongly depends on the nature of R. For R = CH_3, the *endo*-H abstraction predominates; when R = $C_6H_5CH_2$ C—C bond fission and *exo*-R abstraction is major. In some cases (R = C_2H_5, C_5H_5), both the reactions occur.

Id is slowly decomposed by 0.1 *N* HCl, the products being a mixture of IId and III, and tars due to complete disintegration of the π-complex. The cyclohexadienyl compounds are also to a certain degree decomposed when put in contact with methylene chloride for a longer time (two hours).

Finally, the iron derivatives Ib-d are in part transformed to the arene(cyclopentadienyl)iron cations when chromatographed on thin layers of alumina (GOST activity 2) not protected from the air. Reaction 4 usually applies in this case and a mixture of the substituted and unsubstituted cations is formed. With Ic, the *exo*-benzyl radical is abstracted to result in III only. Thermolysis of cyclohexadienyl(cyclopentadienyl)iron derivatives leads to ferrocene. The latter formed even from the most stable, unsubstituted species, $C_6H_7FeC_5H_5$, when treated at 110-115° for 1.5 h.

Experimental

All operations were carried out in a well-purified argon or nitrogen atmosphere, in absolute solvents free of atmospheric oxygen. Compounds Ia-Id were not contaminated with $C_6H_7FeC_5H_5$, as verified by the absence of the 2800 cm^{-1} band (the *exo*-H absorption) from the IR spectra.

1. Oxidation of (cyclohexadienyl)cyclopentadienyliron derivatives with $(Ph_3C)BF_4$

A solution of $(Ph_3C)BF_4$ and a (cyclohexadienyl)cyclopentadienyliron derivative in CH_2Cl_2 was stirred by an inert gas sweep at room temperature for 30 min. The red colour of the starting iron compound gradually paled, transformed to yellow, and the solution became opaque. The solvent was evaporated in vacuo, the residue was treated with water, the yellow solution in water was washed with ether and evaporated to dryness in vacuo at 30-40°. The resulting (arene)cyclopentadienyliron tetrafluoroborates were extracted with methylene dichloride and analysed on thin layers of alumina (dichloroethane/ethanol 25/4 v/v, or dichloroethane/iso-C_3H_7OH 3/1). The plates were developed in iodine vapour. Under these conditions $[C_6H_6FeC_5H_5]^+$ forms a slowly moving, violet-brown spot readily discernible from the yellow, much more mobile spots of the cations carrying hydrocarbon substituents. Authentic samples were applied wherever possible. Preparative separations were accomplished on thin layers of alumina in the same solvent systems.

When the cation mixture could not be separated it was pyrolysed (200-250°, 30 min) and the resulting arenes were analysed by gas-liquid chromatography.

2. Oxidation of (cyclohexadienyl)cyclopentadienyliron derivatives with bromosuccinimide

Bromosuccinimide (BS) was added to a solution of a (cyclohexadienyl)-cyclopentadienyliron derivative in a 1/5 mixture of light petroleum and methanol. The reaction mixture was stirred by an inert gas sweep at room temperature for 30 min, and a solution of $NaPF_6$ in methanol was gradually added. The mixture was filtered and evaporated in vacuo. Further treatment, analysis, and separation are similar to those in *1*.

All (cyclohexadienyl)cyclopentadienyliron compounds were oxidised using procedures *1* and *2*. Below, just the results are reported.

3. *Oxidation of (exo-ethylcyclohexadienyl)cyclopentadienyliron (Ib)*

The oxidation of *exo*-Ib with either $(Ph_3C)BF_4$ or BS results, as a rule, in a mixture of $[C_6H_6FeC_5H_5]X$ and $[C_2H_5C_6H_5FeC_5H_5]X$ (X is BF_4^- or PF_6^-) in which $[C_6H_6FeC_5H_5]X$ prevails. Occasionally, $[C_6H_6FeC_5H_5]X$ alone may be formed. Lowering the reaction temperature does not suppress the C_2H_5 abstraction. The cation mixture could not be separated either chromatographically or by repeated crystallisations. Table 1 lists results of qualitative chromatographical analyses and GLC data for the pyrolysis products.

4. *Oxidation of (exo-benzylcyclohexadienyl)cyclopentadienyliron (exo-Ic)*

Action of BS. *exo*-Ic reacts with BS to give the unsubstituted $[C_6H_6FeC_5H_5]^+$ in all cases. For example *exo*-Ic (2 mmol) with BS (4 mmol) gave $[C_6H_6FeC_5H_5]PF_6$, 0.25 g (35%), identified chromatographically and analysed. (Found: C, 38.29; H, 3.20: $C_{11}H_{11}F_6FeP$ calcd.: C, 38.40; H, 3.22%.) The experiment was carried out repeatedly. The maximal yield was 50%.

Action of $(Ph_3C)BF_4$. *exo*-Ic (3.5 mmol) and $(Ph_3C)BF_4$ (3.6 mmol) gave 1.61 g of a mixture of tetrafluoroborates. The mixture was chromatographed on thin layers of Al_2O_3 to give $[C_6H_6FeC_5H_5]BF_4$, 0.83 g (41%), identified chromatographically, and $[C_6H_5CH_2C_6H_5FeC_5H_5]BF_4$, 0.13 g (5%), m.p. 126-128° (recrystallised from dichloroethane). (Found: C, 57.40; H, 4.60. $C_{18}H_{17}F_4FeB$ calcd.: C, 57.50; H, 4.59%.) With an increase in the relative amount of $(Ph_3C)BF_4$ the unsubstituted cation becomes the only product. The yield of $[C_6H_6FeC_5H_5]BF_4$ is 68% at the $(Ph_3C)BF_4$/Ic ratio of 2/1, it is just 24% at the ratio of 4/1 since strong decomposition starts under these conditions.

Oxidation on alumina. When Ic (5 mmol) was chromatographed on a thin layer of alumina (GOST activity 2) not protected from atmospheric oxygen (the eluent was heptane), the crimson colour of the cyclohexadienyl compound gave way to the yellow colour of the cation*. The yellow band, immobile in heptane, was eluted by dichloroethane/ethanol (25/4). The solvent was removed, the residue dissolved in water, treated with $NaBF_4$, and the solution was worked up in the usual way to give 0.9 g (30%) of $[C_6H_6FeC_5H_5]$-BF_4 which was converted into the tetraphenylborate and analysed. (Found: C, 81.34; H, 6.04; Fe, 10.76. $C_{35}H_{31}BFe$ calcd.: C, 81.10; H, 6.03; Fe, 10.77%.)

5. *Oxidation of exo-cyclopentadienylcyclohexadienyl(cyclopentadienyl)iron (exo-Id)*

By $(Ph_3C)BF_4$. *exo*-Id (3.0 mmol) reacts with $(Ph_3C)BF_4$ (3 mmol) to give a mixture (0.6 g) of tetrafluoroborates of the cations. The mixture was chromatographed on thin layers of alumina to give $[C_6H_6FeC_5H_5]BF_4$ (identified chromatographically), 0.15 g (16%), and $[C_5H_5C_6H_5FeC_5H_5]BF_4$, 0.3 g (27%)). The latter compound, very hygroscopic, was converted into the hexafluorophosphate, m.p. 157-160°, before analysis. (Found: C, 46.94; H, 4.25; F, 27.33; Fe, 13.55. $C_{16}H_{15}F_6FeP$ calcd.: C, 47.17; H, 3.71; F, 27.94; Fe, 13.69%.) Also, the tetraphenylborate $[C_5H_5C_6H_5FeC_5H_5]BPh_4$, m.p. 180-183°, was isolated.

The experiment carried out repeatedly at a $(Ph_3C)BF_4$/*exo*-Id ratio of 1/1 or 2/1 led to identical results in all cases: $[C_6H_6FeC_5H_5]BF_4$, 10 to 16%,

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* The anion is probably OH^- adsorbed on Al_2O_3 [15].

TABLE 1

OXIDATION OF *exo*-ETHYLCYCLOHEXADIENYL(CYCLOPENTADIENYL)IRON

Amount of Ib (g), and the oxidant/Ib ratio	Products detected by chromatography on Al_2O_3	Total yield of salts [a] g (%)	Results of GLC analysis
Reaction with $(Ph_3C)BF_4$			
0.60 (1/1)	$[C_6H_6FeC_5H_5]BF_4 + [C_2H_5C_6H_5FeC_5H_5]BF_4$	0.25 (40)	benzene + ethylbenzene
0.42 (2/1)	$[C_6H_6FeC_5H_5]BF_4 + [C_2H_5C_6H_5FeC_5H_5]BF_4$	0.21 (33)	benzene + ethylbenzene
0.35 (3/1)	$[C_6H_6FeC_5H_5]BF_4 + [C_2H_5C_6H_5FeC_5H_5]BF_4$	0.13 (40)	beznene + traces of ethylbenzene
Reaction with BS			
0.28 (1/1)	$[C_6H_6FeC_5H_5]PF_6 + [C_2H_5C_6H_5FeC_5H_5]PF_6$	0.30 (75)	benzene + ethylbenzene
0.25 (2/1)	$[C_6H_6FeC_5H_5]PF_6 + [C_2H_5C_6H_5FeC_5H_5]PF_6$	0.24 (80)	benzene + ethylbenzene
0.70 (2/1)	$[C_6H_6FeC_5H_5]PF_6 + [C_2H_5C_6H_5FeC_5H_5]PF_6$	0.30 (33)	benzene + traces of ethylbenzene
0.30 (2/1)	$[C_6H_6FeC_5H_5]PF_6$	0.20 (50)	benzene

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[a] The yield is calculated in terms of $(C_6H_6FeC_5H_5)X$. Although they are not quite accurate (the mixtures contain also $[C_2H_5C_6H_5FeC_5H_5]X$) the data allow one to estimate the amount of the starting compound oxidised to the cation.

and $[C_5H_5C_6H_5FeC_5H_5]BF_4$, 20 to 27%. The yield in terms of the mixture before separation was as high as 50 to 60%.

By BS. *exo*-Id (9 mmol) with BS (18 mmol) gave 1.6 g of a mixture of the hexafluorophosphates. The mixture was separated chromatographically on thin layers of alumina to give $[C_6H_5FeC_5H_5]PF_6$, 0.12 g (3%), analysed in the form of tetraphenylborate. (Found: Fe, 10.97. $C_{35}H_{31}BFe$ calcd.: Fe, 10.77%.) Another bromine-containing cation (0.7 g of the hexafluorophosphate) was also obtained. Its analysis agrees with the structure resulting from addition of an HBr molecule to $[C_5H_5C_6H_5FeC_5H_5]^+$. The decomposition point is 146-148° after recrystallisation from ethanol. (Found: C, 39.51; H, 3.26; F, 23.38; Fe, 11.84. $C_{16}H_{16}BrPF_6Fe$ calcd.: C, 39.26; H, 3.29; F, 23.30; Fe, 11.42%.)

The tetraphenylborate of the presumed $[BrC_5H_6C_6H_5FeC_5H_5]^+$ decomposes at 172-173° after recrystallisation from acetonitrile.

6. *Oxidation of (methylcyclohexadienyl)cyclopentadienyliron isomers*

Oxidation of (exo-methylcyclohexadienyl)cyclopentadienyliron (exo-Ia). *exo*-Ia (1.4 mmol) with $(Ph_3C)BF_4$ (1.2 mmol) gave $[CH_3C_6H_5FeC_5H_5]BF_4$, 0.12 g (30%). Traces of $[C_6H_6FeC_5H_5]BF_4$ were found on the Al_2O_3 chromatogram. GLC analysis of the pyrolysis products showed the presence of toluene and traces of benzene. The synthesis was completely reproducible. Oxidation of exo-Ia with BS leads to the higher amount of the unsubstituted cation, as verified by GLC, revealing toluene and significant amounts of benzene.

Oxidation of (exo-H-methylcyclohexadienyl)cyclopentadienyliron (IV). IV (2.2 mmol) with $(Ph_3C)BF_4$ (2.4 mmol) gave $[CH_3C_6H_5FeC_5H_5]BF_4$, 0.16 g (22%), identified chromatographically on Al_2O_3. Thermolysis resulted in $CH_3C_6H_5$ only (GLC). Benzene admixtures found in some of the experiments may be explained by assuming that the starting IV was contaminated with Ia (see 7). The $[CH_3C_6H_5FeC_5H_5]BF_4$ yield increases with the relative amount of $(Ph_3C)BF_4$ and reaches 54% at a IV/$(Ph_3C)BF_4$ ratio of 1/3. The action of BS led to the same results. The $[CH_3C_6H_5FeC_5H_5]PF_6$ yield was 75% at a IV/BS ratio of 1/2.

7. *Synthesis of (exo-H-methylcyclohexadienyl)cyclopentadienyliron (IV)*

A large excess of $LiAlH_4$ was added to a suspension of $[CH_3C_6H_5FeC_5H_5]PF_6$ (2 g, 8 mmol) in 80 ml dimethoxyethane. The mixture was stirred at room temperature for two hours. The solution turned red. Ether, methanol, and water were carefully added, the mixture was filtered, the layers were separated, and the aqueous layer was washed with ether. The combined ether solutions resulted in a red oil which was distilled in vacuo to give IV, 0.7 g (42%), b.p. 80-85°/0.5 mm. (Found: C, 67.27; H, 6.69; Fe, 25.25. $C_{12}H_{14}Fe$ calcd.: C, 67.28; H, 6.54; Fe, 26.16%.) IR spectra of IV contain a band at 2780 cm^{-1}, indicating the presence of an *exo*-H atom. The PMR spectrum suggests that the compound is a mixture of isomers differing in the position of CH_3 and containing, inter alia, Ia as admixture. PMR spectra of Ia-Id and IV have been published elsewhere.

8. *Check-up experiments*

Action of oxygen. A solution of Id (0.05 g, 0.18 mmol) in 3 ml light petroleum plus 15 ml methanol containing 0.05 g $NaPF_6$ was stirred by a sweep of the air for 2.5 h. The red solution turned yellow. The water was added, the layers were separated, the aqueous layer was evaporated to result in a mixture of hexafluorophosphates (0.018 g) containing (TLC on Al_2O_3) $[C_6H_6FeC_5H_5]PF_6$ and $[C_5H_5C_6H_5FeC_5H_5]PF_6$. Also, products of the complete decomposition, inorganic iron compounds, were observed. Under these conditions the compound Ic gave just $[C_6H_6FeC_5H_5]PF_6$ and the decomposition products.

Action of methylene dichloride. A solution of Id (0.05 g, 0.18 mmol) in 30 ml CH_2Cl_2 was stirred by a nitrogen sweep at room temperature for 2 h. The colour remained almost unchanged, and a thin precipitate was formed. The solvent was removed, ether and a solution of 0.02 g $NaPF_6$ in 20 ml water were added. The layers were separated, the aqueous layer gave 0.008 g of a mixture of $[C_6H_6FeC_5H_5]PF_6$ and $[C_5H_5C_6H_5FeC_5H_5]PF_6$ (TLC on Al_2O_3). From the ether layer Id (0.035 g, 70%) was isolated. The unsubstituted $C_6H_7FeC_5H_5$ is stable against CH_2Cl_2. Salts of (arene)cyclopentadienyliron cations are stable as well under these conditions. Special experiments showed that using CH_2Cl_2 as solvent virtually did not affect the results of the oxidations involving $(Ph_3C)BF_4$. Check-up oxidations of Id were made in CH_2Cl_2 and in acetonitrile. The results were practically identical.

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