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THE SYNTHESIS OF SOME COMPLEXED KETONES OR SULFONES BY OXIDATION OF η^6-ARENE-η^5-CYCLOPENTADIENYLIRON CATIONS OR η^6-ARENEDI-η^5-CYCLOPENTADIENYLIRON DICATIONS

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Summary

Di-η^5-cyclopentadienyliron complexed dications of 9,10-dihydroanthracene, xanthene, thioxanthene and diphenylmethane derived from ligand exchange reactions but without their prior isolation were oxidized with $KMnO_4$ to give, respectively, the dications of anthraquinone, xanthone, thioxanthone and benzophenone, isolated as their dihexafluorophosphate salts. Cyclopentadienyliron complexes of arenes containing a sulfide function were oxidized by *m*-chloroperbenzoic acid to the corresponding complexed sulfones, and sulfones prepared in this way include the hexafluorophosphate salt of the η^6-*p*-tolylsulfonylbenzene, thioxanthene-10,10-dioxide, 9,9-dimethylthioxanthene-10,10-dioxide or dibenzothiophene-9,9-dioxide-η^5-cyclopentadienyliron cation.

Introduction

Direct $AlCl_3$/Al catalyzed ligand exchange between ferrocene and ketoarenes has not been successfully carried out, presumably because the mechanism of the ligand exchange could involve steps similar to electrophilic aromatic substitution [1,2] and this would be difficult with arenes containing an electron-withdrawing keto substituent. In this laboratory, it was found that η^6-ketoarene-η^5-cyclopentadienyliron cations could be prepared from the $KMnO_4$ oxidation of the aqueous solution of the tetrachloroaluminate salts of η^6-arene-η^5-cyclopentadienyliron cations derived from ligand exchange reactions without the prior isolation of the product from the ligand exchange [3,4]. Such an in situ oxidation could convert a methylene group directly bonded to two aromatic rings in a cyclopentadienyliron (CpFe) complexed arene to a carbonyl function, thus giving rise to the CpFe complexes of benzophenone, fluorenone, anthraquinone, xanthone and thioxanthone from the oxidation of the corresponding CpFe complexes of diphenylmethane, fluorene, 9,10-dihydroanthracene, xanthene and thioxanthene [3,4]. The

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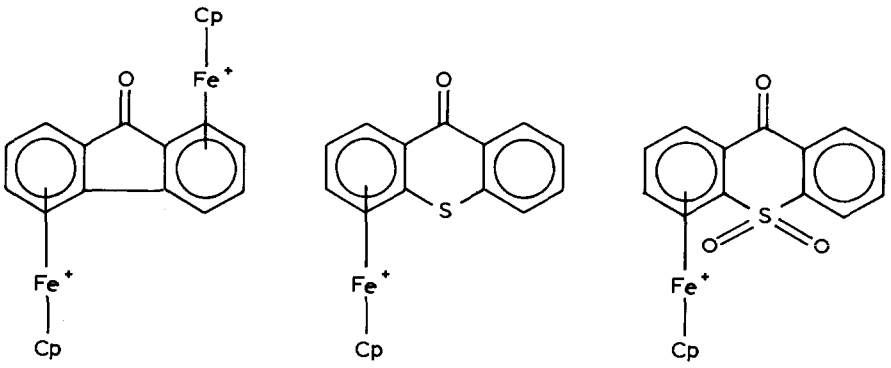
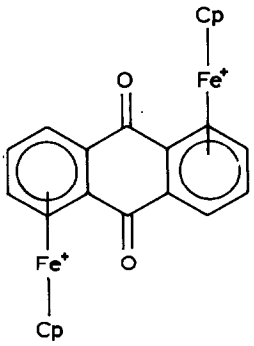


oxidation of the η^6-fluorene-*trans*-di-η^5-cyclopentadienyliron dication was also found to give the η^6-fluorenone-*trans*-di-η^5-cyclopentadienyliron dication (I) [3]. In the present work, some additional di-η^5-cyclopentadienyliron dications of ketoarenes are prepared. Moreover, in the $KMnO_4$ oxidation of the η^6-thioxanthene-η^5-cyclopentadienyliron cation, beside the thioxanthone complex (II), oxidation of the sulfide function to the sulfone also took place to give a mixture of II and the η^6-thioxanthen-9-one-10,10-dioxide-η^5-cyclopentadienyliron cation (III) [4]. In the present paper, the selective oxidation of complexed sulfides to sulfones without oxidizing a methylene group to the ketone is also described.

Results and discussion

Using the same procedure as in the preparation of dication I from the in situ $KMnO_4$ oxidation of the di-η^5-cyclopentadienyliron complexed fluorene [3], ligand exchange between an excess of ferrocene and $AlCl_3$/Al with 9,10-dihydroanthracene, xanthene, thioxanthene or diphenylmethane gave the di-η^5-cyclopentadienyliron complexed dication of 9,10-dihydroanthracene [5], xanthene [6,7] thioxanthene [6,7], or diphenylmethane [8], respectively, and, without prior isolation of these dications, treatment with $KMnO_4$ gave the corresponding di-η^5-cyclopentadienyliron complexed ketoarenes, namely, the η^6-anthraquinone, xanthone, thioxanthone or benzophenonedi-η^5-cyclopentadienyliron dication (IV, V, VI or VII, respectively). For IV, V and VI, the two CpFe groups are assigned the *trans* configuration since their precursor arene dications were shown to be *trans* [5–7].

Dications IV, V and VI were isolated as the dihexafluorophosphate salts. It was noted from the ^1H NMR spectra that, in the case of V, about 10% of the product was the monocation with the ketoarene complexed only to one CpFe group. This monocation, however, could be removed by washing with a 5/1 mixture of acetone/CH_2Cl_2, leaving the pure dihexafluorophosphate salt of dication V. As a possible explanation for the presence of the monocation, it may be suggested that during the ligand exchange, beside dimetallation to give the dication, there may be the formation of a minor amount of the monocation with complexation only to one CpFe group, and this possibility has been pointed out [6]. In the isolation and



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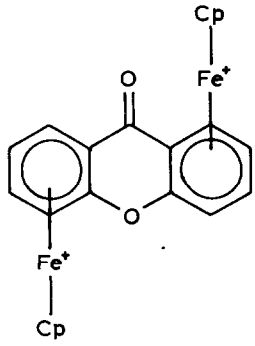

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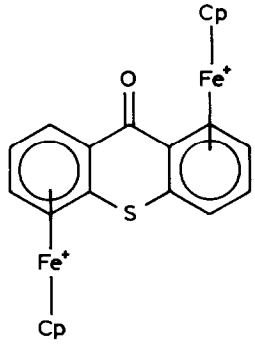

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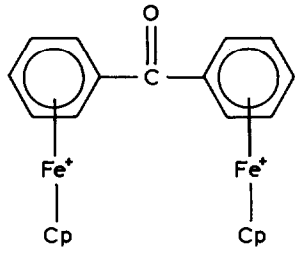


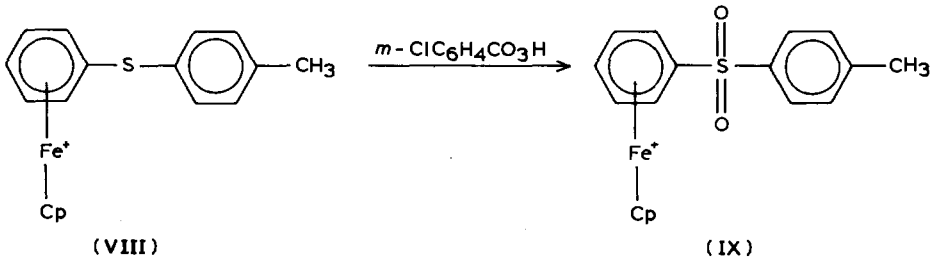
(VII)

purification of the dihexafluorophosphate of the dication of xanthene [6], any minor amount of monometallated xanthene would have been effectively removed. In the present in situ oxidation, however, $KMnO_4$ was added to the aqueous solution of the ligand exchange products without their isolation, and the minor amount of monometallated xanthene would also be oxidized to the corresponding complexed ketoarene monocation. This explanation was verified by showing that when the ligand exchange between xanthene and an excess of ferrocene in the presence of $AlCl_3/Al$ was carried out, the crude product actually contained both the mono- and di-cations.

The oxidation of η^6-diphenylmethanedi-η^5-cyclopentadienyliron dication to the corresponding benzophenone dication VII presented some difficulties in that complete oxidation could not be attained. In spite of changes in reaction time or in the amount of $KMnO_4$ employed, the products obtained were an approximately 1/1 mixture of the dihexafluorophosphate salts of VII and the unreacted diphenylmethane dication and these were difficult to separate. A yield of VII, therefore, was estimated from the total mixed products, half of which being VII. The reason for the failure to give complete oxidation to VII is not clear. However, it may be pointed out that in the oxidation to give I [3] and IV, V and VI, all the precursor dications have the two CpFe groups held in the *trans*-configuration. Only in the oxidation to give VII, can the complexed diphenylmethane dication have the two CpFe com-

plexed benzene rings freely rotating, and this may provide some steric hindrance when the two CpFe groups are *cis* or situated on the same side, thus blocking an easy approach by the oxidizing agent. By repeatedly dissolving the product mixture in acetone followed by careful precipitation with ether, more and more of the diphenylmethane dication was removed and a small, fairly pure sample of VII as its dihexafluorophosphate was obtained for elemental analysis and spectral studies. The yields, analyses and spectral data for dications IV, V, VI and VII are summarized in Tables 1, 2 and 3.

m-Chloroperbenzoic acid (*m*-CPBA) has been utilized in oxidations of sulfides to sulfoxides and sulfones [9], and a recent application involved the oxidation of chiral sulfides containing a ferrocenyl group to chiral sulfoxides and sulfones [10]. In the present work, it was found that CpFe complexes of arenes containing a sulfide function could be oxidized by *m*-CPBA under mild conditions to give the corresponding complexed sulfone without contamination by the sulfoxide. Thus a CH_2Cl_2 solution of the hexafluorophosphate salt of the η^6-*p*-tolylthiobenzene-η^5-cyclopentadienyliron cation (VIII) [11] when stirred with an excess of *m*-CPBA at room temperature for 4 h gave an 85% yield of the hexafluorophosphate of the η^6-*p*-tolylsulfonylbenzene-η^5-cyclopentadienyliron cation (the complexed phenyl *p*-tolyl sulfone) (IX). Other reagents such as H_2O_2 in CH_3COOH [12] and $H_2O_2/TiCl_3$ [13] were found to be less effective than *m*-CPBA and gave much lower yields of an impure complexed sulfone which was more difficult to purify.



(VIII) (IX)

It was indicated earlier that the in situ $KMnO_4$ oxidation of the η^6-thioxanthene-η^5-cyclopentadienyliron cation (X) gave a mixture of the CpFe complexes of thioxanthone (II) and thioxanthen-9-one-10,10-dioxide (III) [4]. When the thioxanthene complex X was treated with *m*-CPBA, the η^6-thioxanthene-10,10-dioxide-η^5-cyclopentadienyliron cation (XI) was obtained; thus only the sulfide function was oxidized to the sulfone, with the C(9) methylene group unaffected. Deprotonation of a CpFe complex at an α-carbon position has been shown to give a zwitterionic species that could act as a nucleophile in substitution reactions [14]. When the thioxanthene complex X was deprotonated with t-BuOK followed by treatment with an excess of CH_3I, dimethylation at C(9) took place to give the η^6-9,9-dimethylthioxanthene-η^5-cyclopentadienyliron cation (XII), which on oxidation with *m*-CPBA gave the corresponding complexed sulfone, the η^6-9,9-dimethylthioxanthene-10,10-dioxide-η^5-cyclopentadienyliron cation (XIII). Interestingly, XIII could also be prepared in an alternative way via the deprotonation of XI followed by treatment with CH_3I.

When the η^6-dibenzothiophene-η^5-cyclopentadienyliron cation (XIV) [6] was oxidized with *m*-CPBA, besides the expected η^6-dibenzothiophene-9,9-dioxide-η^5-

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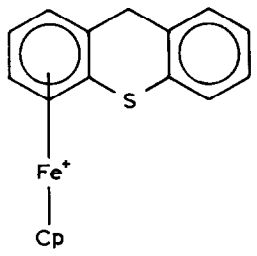

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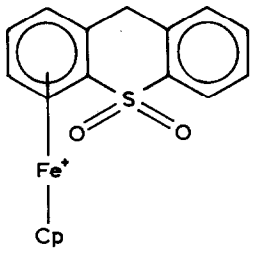

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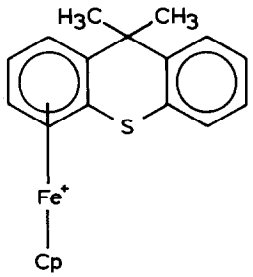

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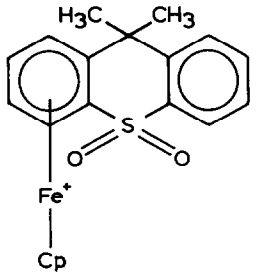


(XIII)

TABLE 1

YIELDS AND ANALYTICAL DATA FOR η^6-KETOARENEDI-η^5-CYCLOPENTADIENYLIRON DIHEXAFLUOROPHOSPHATES AND FOR η^6-SULFONYLARENE-η^5-CYCLOPENTADIENYLIRON HEXAFLUOROPHOSPHATES

Complex	Yield (%)	Analysis (Found (calcd.) (%))	
		C	H
trans-(Anthraquinone)(CpFe)$_2$(PF$_6$)$_2$, IV-(PF$_6$)$_2$	51	38.47 (38.95)	2.45 (2.45)
trans-(Xanthone)(CpFe)$_2$(PF$_6$)$_2$, V-(PF$_6$)$_2$	20	37.18 (37.95)	2.47 (2.49)
trans-(Thioxanthone)(CpFe)$_2$(PF$_6$)$_2$, VI-(PF$_6$)$_2$	60	36.93 (37.13)	2.17 (2.44)
(Benzophenone)(CpFe)$_2$(PF$_6$)$_2$, VII-(PF$_6$)$_2$	18	37.58 [a] (38.69)	2.10 [a] (2.82)
(*p*-CH$_3$C$_6$H$_4$SO$_2$C$_6$H$_5$)CpFePF$_6$, IX-PF$_6$	85	42.70 (43.40)	3.43 (3.44)
(Thioxanthene-10,10-dioxide)CpFePF$_6$, XI-PF$_6$	85	42.72 (43.57)	3.35 (3.04)
(9,9-Dimethylthioxanthene)CpFePF$_6$, XII-PF$_6$ [b]	81	48.29 (48.80)	3.78 (3.89)
(9,9-Dimethylthioxanthene-10,10-dioxide)CpFePF$_6$, XIII-PF$_6$	80	45.85 (45.82)	3.60 (3.64)
(Dibenzothiophene-9,9-dioxide) CpFePF$_6$, XV-PF$_6$	40	43.47 [c] (42.35)	3.12 [c] (2.72)

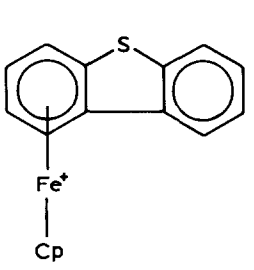
[a] Contaminated by some unoxidized reactant, (diphenylmethane)(CpFe)$_2$(PF$_6$)$_2$. [b] Precursor to the sulfone complex XIII. [c] Contaminated by some unoxidized reactant, (dibenzothiophene)CpFePF$_6$.

TABLE 2

DATA FROM THE ^{1}H NMR AND IR SPECTRA OF KETOARENE AND SULFONYLARENE COMPLEXES

Complex	δ (ppm from TMS)					IR (cm^{-1})
	Solvent	Cp	Complexed Ar	Uncomplexed Ar	Others	
IV-$(PF_6)_2$	$(CD_3)_2SO$	5.43(s,10H)	7.2–7.5(m,8H)			1695(CO)
V-$(PF_6)_2$	CD_3NO_2	5.23(s,10H)	6.5–7.4(m,8H)			1710(CO)
VI-$(PF_6)_2$	CD_3NO_2	5.20(s,10H)	6.4–7.4(m,8H)			1685(CO)
VII-$(PF_6)_2$	CD_3CN	5.09(s,10H)	6.6–6.8(m,10H)			1700(CO)
IX-PF_6	$(CD_3)_2CO$	5.46(s,5H)	6.6–7.2(m,5H)	7.49,8.03(2d,4H)	2.43(s,3H,CH_3)	1150,1325(SO_2)
XI-PF_6	$(CD_3)_2CO$	4.86(s,5H)	6.6–7.4(m,4H)	7.6–8.1(m,4H)	4.56(s,2H,CH_2)	1152,1320(SO_2)
XII-PF_6 [a]	$(CD_3)_2CO$	4.69(s,5H)	6.4–7.0(m,4H)	7.4–8.0(m,4H)	1.49,2.64(2s,6H,2CH_3)	
XIII-PF_6	CD_3NO_2	4.89(s,5H)	6.5–7.0(m,4H)	7.4–8.0(m,4H)	1.89,2.33(2s,6H,2CH_3)	1150,1320(SO_2)
XV-PF_6	$(CD_3)_2CO$	5.30(s,5H)	6.6–7.0(m,4H)	7.4–8.4(m,4H)		1160,1310(SO_2)

[a] Precursor to XIII.



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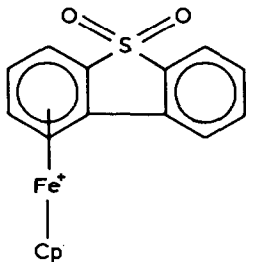
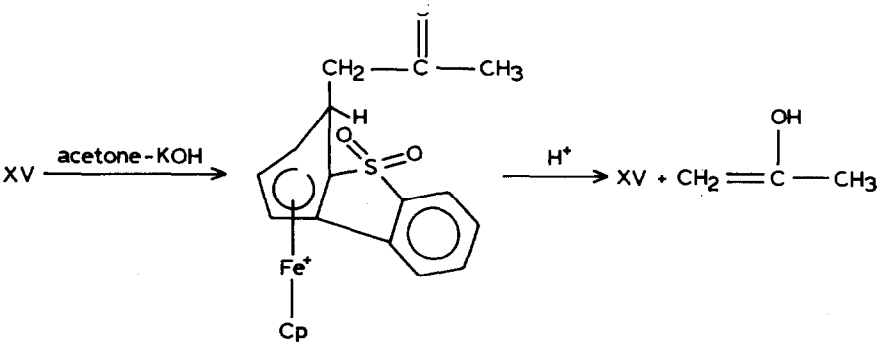


(XV)

cyclopentdienyliron cation (XV), considerable amounts of unreacted XIV remained as a contaminant. In order to remove the contaminant, the mixture was treated with acetone in aqueous KOH to give the Yanovsky (Janovsky) adduct XVI derived from addition of the acetonyl anion to XV [15]. Adduct XVI could then be separated from the unreacted cation XIV by $CHCl_3$ extraction, and upon treatment of XVI with HCl, a reversal of the Yanovsky reaction generated cation XV which was isolated as its hexafluorophosphate with an overall yield of 40%. The yields and analytical and spectral data for complexed sulfones IX, XI, XIII and XV, as well as complex XII, the precursor to XIII, are included in Tables 1–3.

Finally, it may be recalled that the in situ $KMnO_4$ oxidation of the η^6-thioxanthene-*trans*-di-η^5-cyclopentadienyliron dication (XVII) gave the thioxanthone dication VI with the sulfide function unaffected. Similarly, when dication XVII or the η^6-dibenzothiophene-*trans*-di-η^5-cyclopentadienyliron dication (XVIII) [6] was treated with *m*-CPBA, no reaction took place and the starting material, XVII or XVIII, could be recovered in high yield. Thus in contrast to the ready oxidation of the sulfide to sulfone in the monometallated CpFe complex of thioxanthene or dibenzothiophene, analogous *trans*-dimetallated S-containing heterocyclic systems complexed to two CpFe groups failed to give any oxidation of the sulfide to sulfone. The reason for this failure, however, is not clear, though possibly steric hindrance may play a role.



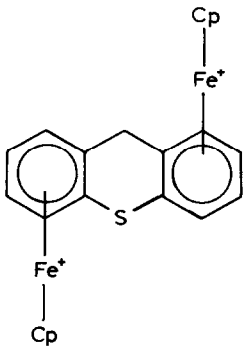
(XVI)

TABLE 3

DATA FROM THE ^{13}C NMR SPECTRA OF KETOARENE AND SULFONYLARENE COMPLEXES

Complex	δ (ppm from TMS) [a]				
	Solvent	Cp	Complexed Ar	Uncomplexed Ar	Others
IV-$(PF_6)_2$	$(CD_3)_2SO$	80.9	87.4,92.0,87.9*		184.6(CO)
V-$(PF_6)_2$	CD_3NO_2	81.2	80.4,84.1,89.4, 91.4,78.2*,131.5*		183.4(CO)
VI-$(PF_6)_2$	$(CD_3)_2SO$	79.5	81.5,84.8,87.8, 88.9,81.7*,105.1*		184.1(CO)
VII-$(PF_6)_2$	$(CD_3)_2SO$	78.8	88.4,89.0,90.2 94.7*		194.5(CO)
IX-PF_6	$(CD_3)_2CO$	80.3	88.2,89.7,91.3, 107.7*	129.2,131.6, 136.7*,147.6*	21.6(CH_3)
XI-PF_6	$(CD_3)_2CO$	80.3	83.9,88.5,89.6, 90.4,103.6*,104.0*	124.8,130.3,131.1, 135.4,136.9*,137.8*	33.2(CH_2)
XII-PF_2 [b]	$(CD_3)_2CO$	78.1	84.0,86.7,87.6, 89.3,104.1*,108.8*	126.2,128.6,128.9 129.8,130.9*,142.0*	24.3,24.9($2CH_3$) 41.0*(C(9))
XIII-PF_6	CD_3NO_2	80.7	84.7,85.5,89.3, 90.7,102.7*,112.4*	126.1,127.7,130.7 136.2,136.4*,141.5*	25.4,34.9($2CH_3$) 41.0*(C(9))
XV-PF_6	CD_3NO_2	82.5	82.6,83.6,88.3, 90.9,95.6*,102.9*	124.1,125.7,134.9, 137.0,129.5*,139.1*	

[a] Asterisks denote quaternary carbons. [b] Precursor to XIII.



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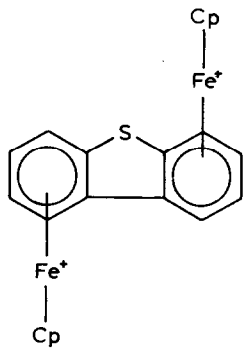

(XVIII)

Experimental

η^6-Anthraquinone-trans-di-η^5-cyclopentadienyliron dihexafluorophosphate (IV-PF_6)$_2$

A mixture of 3.60 g (20 mmol) of 9,10-dihydroanthracene, 18.6 g (100 mmol) of ferrocene, 32.0 g (240 mmol) of $AlCl_3$ and 0.80 g (30 mmol) of Al powder in 70 ml of decalin under an atmosphere of N_2 was heated with stirring under reflux at 140 ± 2°C for 6 h. The resulting material was poured into 300 ml of ice-H_2O and the mixture was washed with ether (3 × 50 ml). A solution of 15.8 g (100 mmol) of $KMnO_4$ in 150 ml of H_2O was added and this reaction mixture was heated with stirring at 60°C for 10 h. The precipitated MnO_2 was removed by filtration under

reduced pressure through a bed of Ottawa sand on a sinter glass filter. Upon addition of 6.5 g (40 mmol) of NH_4PF_6 to the filtrate, the product precipitated as an orange-red solid which was purified by dissolution in CH_3NO_3 and reprecipitation with the addition of ether to give 7.5 g (51%) of the dihexafluorophosphate salt of dication IV.

η^6-Xanthone or thioxanthone-trans-di-η^5-cyclopentadienyliron dihexafluorophosphate (V-$(PF_6)_2$ or VI-$(PF_6)_2$)

Using the same procedure as described above for the preparation of IV-$(PF_6)_2$, ligand exchange between xanthene or thioxanthene with an excess of ferrocene followed by $KMnO_4$ oxidation gave the dihexafluorophosphate salt of dication V or VI. In the preparation of VI-$(PF_6)_2$, the only modification in the procedure was that after the introduction of the $KMnO_4$ to the ligand exchange product, the reaction mixture was heated at 60°C for 15 h instead of 10 h. In the preparation of V-$(PF_6)_2$, the final solid product was washed with a 5/1 mixture of acetone/CH_2Cl_2 to remove the small amount of η^6-xanthone-η^5-cyclopentadienyliron hexafluorophosphate, leaving behind a relatively low yield of 20% of the pure V-$(PF_6)_2$.

η^6-Benzophenonedi-η^5-cyclopentadienyliron dihexafluorophosphate (VII-$(PF_6)_2$)

The dihexafluorophosphate salt of the benzophenone dication VII was prepared in the same way as described for the preparation of IV-$(PF_6)_2$, but the product was an approximately 1/1 mixture of VII-$(PF_6)_2$ and the unoxidized η^6-diphenylmethanedi-η^5-cyclopentadienyliron dihexafluorophosphate. Increasing the amount of $KMnO_4$ or extending the reaction time for the oxidation did not improve the result but actually gave a lower ratio of VII-$(PF_6)_2$ to unoxidized diphenylmethane complex. When the product mixture was repeatedly dissolved in acetone followed by careful precipitation with ether, more and more of the unoxidized diphenylmethane complex could be removed, leaving a sample of fairly pure VII-$(PF_6)_2$ for elemental and spectral analyses.

η^6-p-Tolylsulfonylbenzene-η^5-cyclopentadienyliron hexafluorophosphate (IX-PF_6)

A solution of 932 mg (2.0 mmol) of η^6-*p*-tolylthiobenzene-η^5-cyclopentadienyliron hexafluorophosphate [11] and 1.035 g (6.0 mmol) of *m*-CPBA (Aldrich Chemical Co.) in 40 ml of CH_2Cl_2 was heated with stirring under reflux for 4 h. The CH_2Cl_2 was then removed by a rotary evaporator and the solid residue was washed with a 2/3 mixture of chloroform/ether, the washings being decanted off. The resulting yellowish product was redissolved in CH_2Cl_2 and reprecipitated with ether to give 850 mg (85%) of IX-PF_6.

η^6-Thioxanthene-10,10-dioxide or 9,9-dimethylthioxanthene-10,10-dioxide-η^5-cyclopentadienyliron hexafluorophosphate (XI-PF_6 or XIII-PF_6)

The hexafluorophosphate salt of cation XI or XIII was prepared by oxidation of the corresponding CpFe complex of thioxanthene (X) or 9,9-dimethylthioxanthene (XII) with *m*-CPBA as described in the preparation of IX-PF_6 except that the reaction time for the oxidation was extended from 4 h to 12 or 18 h, respectively.

η^6-9,9-Dimethylthioxanthene-η^5-cyclopentadienyliron hexafluorophosphate (XII-PF_6)

To a solution of 696 mg (1.5 mmol) or η^6-thioxanthene-η^5-cyclopentadienyliron hexafluorophosphate in 40 ml of tetrahydrofuran and under a N_2 atmosphere, 400

mg (3.5 mmol) of t-BuOK was added. A dark blue color developed rapidly and the mixture was stirred for 5 min and then 2.0 ml of CH_3I was introduced. Stirring at room temperature and under N_2 was continued for 2.5 h and a brown suspension resulted. A solution of 1.5 mmol of NH_4PF_6 in 25 ml of H_2O was added, the mixture was neutralized with 10% HCl and then extracted with CH_2Cl_2 (4×50 ml). The extract was dried over $MgSO_4$ and the solvent was removed by a rotary evaporator. The residual product was redissolved in CH_2Cl_2 and re-precipitated by the addition of ether to give 600 mg (81%) of XII-PF_6.

η^6-Dibenzothiophene-9,9-dioxide-η^5-cyclopentadienyliron hexafluorophosphate (XV-PF_6)

A solution of 900 mg (2.0 mmol) of η^6-dibenzothiophene-η^5-cyclopentadienyliron hexafluorophosphate (XIV-PF_6) in 50 ml of CH_2Cl_2 was heated under reflux with 1.035 g (6.0 mmol) of *m*-CPBA for 16 h and then worked up as described in the preparation of IX-PF_6. According to ^1H NMR, an approximately 1/1 mixture of XV-PF_6 and the unoxidized XIV-PF_6 was obtained. This product mixture was dissolved in 15 ml of acetone, a solution of 1.0 g of KOH in 5.0 ml of H_2O was introduced and the solution was stirred at room temperature for 40 min to give the Yanovsky reaction for cation XV [15]. The resulting dark red solution was extracted with $CHCl_3$ (4×25 ml), the extract washed with H_2O (2×50 ml), dried over $MgSO_4$ and the solvent removed by a rotary evaporator. The residual oil was dissolved in acetone and treated with 10 ml of 10% HCl and 300 mg of NH_4PF_6, regenerating the hexafluorophosphate salt of XV from the Yanovsky adduct. This regenerated product was recovered by extraction with CH_2Cl_2 (4×25 ml), the extract washed with H_2O, dried over $MgSO_4$ and the CH_2Cl_2 removed by a rotary evaporator. Upon dissolution in acetone and reprecipitation by ether, 385 mg (40%) of XV-PF_6 was obtained as a yellow powder.

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