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REACTION OF [α-(TRIMETHYLSILYL)BENZYL]FERROCENES WITH α-FERROCENYLBENZYL CATIONS

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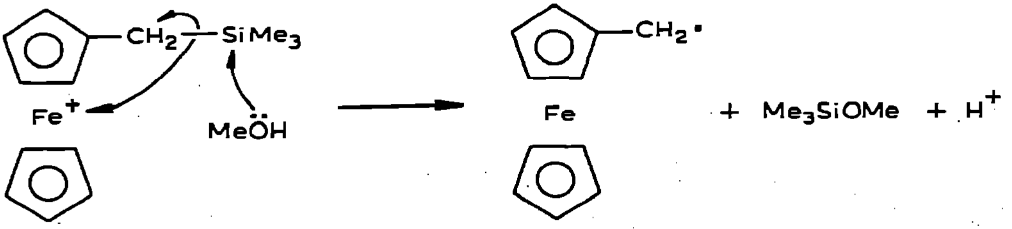
SUMMARY

Although methanolysis of [α-(trimethylsilyl)benzyl]ferrocene (I) and [*p*-methyl-α-(trimethylsilyl)benzyl]ferrocene (II) in the presence of anhydrous ferric chloride merely gave α-ferrocenylbenzyl methyl ether (III) and *p*-methyl-α-ferrocenylbenzyl methyl ether (IV), respectively, acid-catalyzed methanolysis of (I) and (II) in the presence of an equimolar amount of (III) or (IV) afforded 1,2-diferrocenyl-1,2-diarylethanes. It is suggested that one electron oxidation of [α-(trimethylsilyl)benzyl]-ferrocene by α-ferrocenylbenzyl cation generated from α-ferrocenylbenzyl methyl ether, and subsequent methanolysis of the resulting substituted ferricenium ion may occur to give the two species of α-ferrocenylbenzyl radical, which in turn undergo an approximately statistical coupling.

INTRODUCTION

Previously, we have reported an anomalously facile cleavage of the carbon–metal (Si and Ge) bond of ferrocenylmethyl derivatives of silicon and germanium in methanol containing ferric chloride to give ferrocenylmethyl methyl ether[1,2]. A mechanism was postulated in which a substituted ferricenium ion intermediate, produced by a redox reaction of, for example, (ferrocenylmethyl)trimethylsilane with ferric chloride, undergoes ready nucleophilic attack by solvent methanol at silicon presumably with a concomitant intramolecular redox process to give methoxytrimethylsilane and the ferrocenylmethyl radical as shown in eqn. (1). The latter may be rapidly oxidized by a second mole of ferric chloride to give ferrocenylmethyl methyl ether in methanol solution.

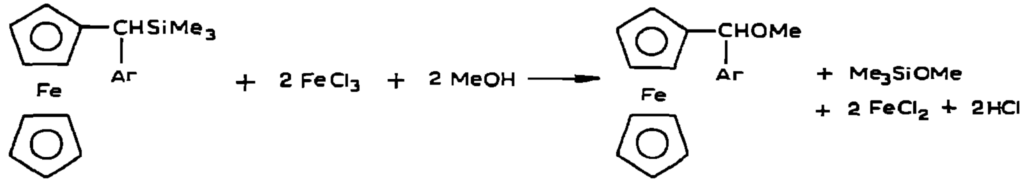


(1)

In this reaction, however, we could not detect any coupling product, *viz.* 1,2-diferrocenylethane, arising from free ferrocenylmethyl radicals. Since the α-ferrocenylbenzyl radical must be more stable, and would tend to dimerize preferably compared to the ferrocenylmethyl radical, if we are concerned with such radical species in the reaction under consideration, the radical coupling may be expected to be observed under suitable conditions. Thus, an investigation of the methanolysis of [α-(trimethylsilyl)benzyl]ferrocene (I) in the presence of ferric chloride was undertaken. It has been found that free radicals are indeed produced from the reaction of (I) with the α-ferrocenylbenzyl cation. The cation was found to have arisen from the corresponding methyl ether under the reaction conditions employed.

RESULTS AND DISCUSSION

The C–Si bonds in [α-(trimethylsilyl)benzyl]ferrocene (I) and [*p*-methyl-α-(trimethylsilyl)benzyl]ferrocene (II) underwent cleavage in a similar fashion to that described previously[1,2], when treated with two equivalents of ferric chloride for each molecule of the substrate in oxygen-free absolute methanol at room temperature, yielding almost quantitatively α-ferrocenylbenzyl methyl ether (III) and *p*-methyl-α-ferrocenylbenzyl methyl ether (IV), respectively [eqn. (2)].



(2)

(I) Ar = Ph
(II) Ar = p-MeC$_6$H$_4$

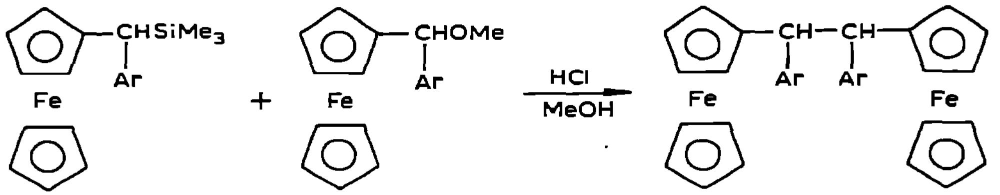
(III) Ar = Ph
(IV) Ar = p-MeC$_6$H$_4$

Furthermore, if compound (I) is treated with an equimolar amount (instead of two equivalents) of ferric chloride at room temperature for 5 min only (III) is produced in 46.1% yield along with unchanged (I) (36.6%), whereas, after a prolonged reaction time, a dimeric product, 1,2-diferrocenyl-1,2-diphenylethane (V), is formed. The latter proved to be a mixture of two diastereomers and was separated by chromatography (alumina column) to give (V_α) (m.p. 218–221°, 60.9% yield) and (V_β) (m.p. 276–278°, 28.7% yield); their assignments to *meso* or *dl* could not be made.

Similar results were obtained for compound (II).

These observations suggest that the coupling occurred between the two different molecules [*viz.*, (I) and (III), and (II) and (IV)] and/or between like individual molecules by the catalytic action of hydrogen chloride formed during methanolysis. In fact, we observed that an equimolar mixture of (I) an (III) [or (II) and (IV)] in a methanol solution of hydrogen chloride (0.01–0.1 *M*) at room temperature gave quantitatively such a dimeric product as shown in eqn. (3).

Moreoever, when (I) and (IV) were allowed to react similarly, a mixture of (V), 1,2-diferrocenyl-1,2-di-*p*-tolylethane (VI), and 1,2-diferrocenyl-1-phenyl-2-*p*-tolylethane (VII) was obtained [eqn. (4); Fc=ferrocenyl]. The percentage composition of these three coupling products was (V) (23%), (VI) (22%), and (VII) (55%) by mass spectrometry.



(3)

(V) Ar = Ph

(VI) Ar = p-MeC$_6$H$_4$

Conversely, reaction of (II) with (III) gave a similar mixture of (V) (23%), (VI) (19%), and (VII) (58%).

$$\begin{array}{l}
\underset{\text{(I)}}{\mathrm{FcCH(Ph)SiMe_3}} + \underset{\text{(IV)}}{\mathrm{FcCH(C_6H_4Me\text{-}}p\mathrm{)OMe}} \xrightarrow[\text{MeOH}]{\text{HCl}} \downarrow \\
\underset{\text{(V)}}{\mathrm{[FcCH(Ph)]_2}} + \underset{\text{(VI)}}{\mathrm{[FcCH(C_6H_4Me\text{-}}p\mathrm{)]_2}} + \underset{\text{(VII)}}{\mathrm{FcCH(Ph)CH(C_6H_4Me\text{-}}p\mathrm{)Fc}} \\
\underset{\text{(II)}}{\mathrm{FcCH(C_6H_4Me\text{-}}p\mathrm{)SiMe_3}} + \underset{\text{(III)}}{\mathrm{FcCH(Ph)OMe}} \xrightarrow[\text{MeOH}]{\text{HCl}} \uparrow
\end{array}$$

The results certainly arise from the fact that two radical species from both [α-(trimethylsilyl)benzyl]ferrocene and α-ferrocenylbenzyl methyl ether were effectively generated and combined statistically.

It has been confirmed that in acidic media an α-ferrocenylbenzyl cation, which is well known to be very stable (see *e.g.* ref. 3), is readily generated from (III) or (IV) as shown by step (*i*) in Scheme 1; namely, both (III) and (IV) were readily converted into the corresponding ethyl ethers when treated with hydrogen chloride in ethanol.

It may be inferred from analogy with one-electron oxidation of ferrocene by triphenylcarbonium ion, which gives ferricenium ion and the triphenylmethyl radical[4], that the resulting α-ferrocenylbenzyl cation oxidizes [α-(trimethylsilyl)-benzyl]ferrocene to a substituted ferricenium ion and α-ferrocenylbenzyl radical [step (*ii*) in Scheme 1]. Generation of α-ferrocenylbenzyl radicals by one-electron reduction of α-ferrocenylbenzyl cations with zinc powder and subsequent radical dimerization have been reported by Cais and Eisenstadt[5].

The resulting substituted ferricenium ion may undergo nucleophilic attack

SCHEME 1

$$\mathrm{FcCH(Ar^1)OMe + H^+ \rightleftarrows FcCH^+Ar^1 + MeOH} \qquad (i)$$

$$\mathrm{FcCH(Ar^2)SiMe_3 + FcCH^+Ar^1 \rightleftarrows Fc^+CH(Ar^2)SiMe_3 + Fc\dot{C}HAr_1} \qquad (ii)$$

$$\mathrm{Fc^+CH(Ar^2)SiMe_3 + MeOH \longrightarrow Fc\dot{C}HAr^2 + Me_3SiOMe + H^+} \qquad (iii)$$

$$\begin{aligned}\mathrm{Fc\dot{C}HAr^1 + Fc\dot{C}HAr^2 \longrightarrow [FcCH(Ar^1)]_2 + [FcCH(Ar^2)]_2 +}\\ \mathrm{+ FcCH(Ar^1)CH(Ar^2)Fc}\end{aligned} \qquad (iv)$$

by methanol at silicon to give the α-ferrocenylbenzyl radical through a concomitant intramolecular redox process as described in the previous papers[1,2] [step (*iii*) in Scheme 1]. Therefore, an approximately statistical combination of the two species of α-ferrocenylbenzyl radicals produced in steps (*ii*) and (*iii*) can occur, as observed, to give the three species of coupling products as shown in Step (*iv*), if step (*iii*) is sufficiently fast.

The mechanism depicted in Scheme 1 may be supported by the fact that (III) [or (IV)] alone was inert under the same reaction conditions. Since such α-ferrocenylbenzyl methyl ethers probably have appreciably greater oxidation potentials than [α-(trimethylsilyl)benzyl]ferrocenes[2,6], only the latter must be oxidized by α-ferrocenylbenzyl cations.

It should be noted that in the reaction of (I) and (II) with ferric chloride in methanol [eqn. (2)], some counter ions such as $Fe_2Cl_6^-$ on the adjoining substituted ferricenium ions may be responsible for a very rapid oxidation of the resultant radical species before the latter behave as typical free radicals.

EXPERIMENTAL

All boiling and melting points are uncorrected. Reactions using organolithium compounds were carried out in an oxygen-free dry nitrogen atmosphere. IR spectra were recorded on a Hitachi EPI G3 grating spectrophotometer. NMR spectra were obtained on a Varian T60 spectrometer in carbon tetrachloride solution with cyclohexane or tetramethylsilane as internal standard and the data are given in τ values. Mass spectra were recorded on a Hitachi RMU 6C mass spectrometer.

p-Methyl-α-ferrocenylbenzyl alcohol[5] and α-ferrocenylbenzyl methyl ether (III)[5,7] were prepared by the method described in the literature.

p-Methyl-α-ferrocenylbenzyl methyl ether (*IV*)

A mixture of 20.0 g (0.0654 mole) of *p*-methyl-α-ferrocenylbenzyl alcohol and 3 ml of acetic acid in 400 ml of methanol was heated at 60° for 1 h. Benzene/hexane (1/1) (300 ml) and water (500 ml) were then added to the cooled reaction mixture. The organic layer was separated and washed with a saturated sodium bicarbonate solution. After evaporation of the solvents, recrystallization of the residue from hexane gave 15.0 g (71.6% yield) of *p*-methyl-α-ferrocenylbenzyl methyl ether (IV), m.p. 120–121° (Found: C, 71.26; H, 6.58. $C_{19}H_{20}FeO$ calcd.: C, 71.27; H, 6.30%). The IR spectrum indicated the presence of methoxy group (2800 cm^{-1}), benzene ring (1613 cm^{-1}), unsubstituted cyclopentadienyl ring (1103 and 998 cm^{-1}), and ether bonding (1077 cm^{-1}). NMR: 7.67 (s, *p*-CH_3), 6.77 (s, OCH_3), 6.02 (bs, C_5H_4 and C_5H_5), 5.15 (s, CH), and 2.85 ppm (bs, C_6H_4).

[*α-(Trimethylsilyl)benzyl*]*ferrocene* (*I*)

To a dispersion of 3.0 g (0.43 g-atom) of lithium in 200 ml of tetrahydrofuran (THF) was added over a period of 20 min a solution of 15.0 g (0.0490 mole) of α-ferrocenylbenzyl methyl ether in 100 ml of THF at −10 to −5°, yielding a deep red solution. After stirring at the same temperature for 1 h, the solution was added dropwise to a solution of 6.5 g (0.060 mole) of trimethylchlorosilane in 50 ml of THF with cooling in an ice bath. The mixture was stirred for 1 h and then hydrolyzed. The resulting organic layer and ether extracts from the aqueous layer were combined, washed with water, dried over anhydrous sodium sulfate, and concentrated *in vacuo*. The petroleum ether-soluble portion of the residue was passed through a column of alumina, evaporated, and fractionally distilled to give 3.5 g (21% yield) of [α-(trimethylsilyl)benzyl]ferricene (I), b.p. 155–157°/0.1 mmHg, m.p. 66–68°. (Found: C, 68.68; H, 7.01. $C_{20}H_{24}FeSi$ calcd.: C, 68.96; H, 6.94%.) IR: 1600, 1247, 1102, and 998 cm^{-1}. NMR: 10.11 [s, $Si(CH_3)_3$], 7.01 (s, CH), 6.28 (s, C_5H_5), 6.03 (s. C_5H_4), and 2.78 ppm (bs, C_6H_5).

[p-Methyl-α-(trimethylsilyl)benzyl]ferrocene (II)

Similarly, a deep red *p*-methyl-α-ferrocenylbenzyllithium solution, prepared from (IV) (11.0 g, 0.0344 mole) and lithium (3.0 g, 0.43 g-atom) in 250 ml of THF at −10 to −5° over a 1-h period, was added to a solution of trimethylchlorosilane (6.0 g, 0.055 mole) in 50 ml of THF with cooling. After work-up as above, 4.5 g (36% yield) of [*p*-methyl-α-(trimethylsilyl)benzyl]ferrocene (II), b.p. 154–156°/0.13 mmHg, m.p. 75–77° was obtained (Found: C, 69.46; H, 7.47. $C_{21}H_{26}FeSi$ calcd.: C, 69.61; H, 7.23%.). IR: 1250, 1107, and 1000 cm^{-1}. NMR: 10.11 [s, $Si(CH_3)_3$], 7.66 (s, *p*-CH_3), 7.06 (s, CH), 6.28 (s, C_5H_5), 6.06 (s, C_5H_4), and 2.96 ppm (s, C_6H_4).

Methanolysis of (I) and (II) in the presence of two equivalents of anhydrous ferric chloride in oxygen-free solution

(*a*). Solutions of 1.12 mg (0.322 mmole) of (I) in 5 ml of absolute methanol and 104 mg (0.644 mmole) of anhydrous ferric chloride in 5 ml of absolute methanol were placed separately in a two-limbed glass tube. The solutions were degassed by several freeze-thawings *in vacuo*. The tube was then sealed with evacuation and the two solutions were mixed at room temperature. The mixture immediately turned greenish blue, indicating the formation of ferricenium ion, and subsequently changed into a clear orange solution in a few minutes. After standing at room temperature for 15 min, the solution was then added to water (50 ml) and the mixture was extracted twice with 30-ml portions of petroleum ether. The extracts were combined, dried over anhydrous sodium sulfate. After evaporation of the solvent, the residue was chromatographed on alumina. Elution with ether gave 78.5 mg (79.6% yield) of (III), m.p. 106–109° (lit.[8] m.p. 111–112°). Its IR and ^1H NMR spectra were identical with those of an authentic sample.

(*b*). Similarly, (II) (126 mg, 0.348 mmole) was allowed to react with $FeCl_3$ (113 mg, 0.700 mmole) in methanol (10 ml) at room temperature for 10 min. After work-up, 89.6 mg (80.5% yield) of (IV) was obtained.

Methanolysis of (I) and (II) in the presence of an equimolar amount of anhydrous ferric chloride in oxygen-free solution

(*a*). In essentially the same way as described above, (I) (215 mg, 0.618 mmole) was reacted with $FeCl_3$ (100 mg, 0.618 mmole) in methanol (10 ml) at room temperature for 5 min. After work-up, chromatography on alumina gave, upon elution with ether, unchanged (I) (78.6 mg, 36.6% yield) and (III) (87.2 mg, 46.1% yield).

In a second run, (I) (116 mg, 0.333 mmole) was treated with $FeCl_3$ (54.0 mg, 0.333 mmole) in methanol (10 ml) at room temperature as above. Yellow crystals gradually precipitated from the reaction mixture after standing for about 10 h. After standing for 87 h, the reaction mixture was dissolved in benzene (100 ml) and the solution was washed with water and dried over anhydrous sodium sulfate. After evaporation of the solvent, the residue was chromatographed on alumina. Elution with ether at first gave 55.8 mg (60.9% yield) of one diastereomer of 1,2-diferrocenyl-1,2-diphenylethane (V_a), m.p. 218–221° (recrystallized from hexane) (lit.[9] m.p. 218–220°). NMR: 6.38 (s, C_5H_5, 12H), 6.18 (bs. C_5H_4, 8 H), and 2.91 ppm (bs, C_6H_5, 10 H). Methine proton resonance probably lies under the cyclopentadienyl resonances. Subsequent elution with benzene gave 26.3 mg (28.7% yield) of the other isomer (V_b), m.p. 277–280° (recrystallized from benzene) (lit.[9] m.p. 276–278°). NMR (in C_6D_6): 6.30 (s, C_5H_5) and 5.92 ppm (bs, C_5H_4). Similarly, methine proton resonance may lie under the cyclopentadienyl resonances and phenyl proton resonances overlapped with the solvent resonances.

(*b*). Compound (II) (123 mg, 0.340 mmole) was treated similarly with $FeCl_3$ (55.0 mg, 0.340 mmole) in methanol (10 ml) at room temperature for 10 min. After work-up, chromatography on alumina as described in (*a*) gave (II) (44.3 mg, 36.0% yield) and (IV) (50.5 mg, 46.4% yield).

In another run, (II) (170 mg, 0.470 mmole) was allowed to react with $FeCl_3$ (76.1 mg, 0.470 mmole) in methanol (10 ml) at room temperature for 120 h and the reaction products were chromatographed on alumina. Elution with ether gave 75.5 mg (55.6% yield) of one diastereomer of 1,2-diferrocenyl-1,2-di-*p*-tolylethane (VI_a), m.p. 223–236° (recrystallized from hexane). (Found: C, 74.07; H, 6.22. $C_{36}H_{34}Fe_2$ calcd.: C, 74.76; H, 5.93%.) NMR: 7.67 (bs, CH_3, 6 H), 6.42 (s, C_5H_5, 12 H), 6.22 (bs, C_5H_4, 8 H), and 3.06 ppm (bs, C_6H_4, 8 H). Elution with benzene gave 38.9 mg (28.6% yield) of the other isomer, m.p. 255–258° (recrystallized from benzene). (Found: C, 74.60; H, 5.87. $C_{36}H_{34}Fe_2$ calcd.: C, 74.76; H, 5.93%.) NMR: 7.63 (bs, CH_3, 6 H), 6.42 (s, C_5H_5, 12 H), and 3.06 ppm (bs, C_6H_4, 8 H). Methine proton resonances may lie under the cyclopentadienyl proton resonances.

Acid-catalyzed coupling reaction of [α-(trimethylsilyl)benzyl] ferrocenes and α-ferrocenylbenzyl methyl ethers in oxygen-free methanol

(*a*). In a two-limbed glass tube were placed separately two solutions, one consisting of 61.0 mg (0.175 mmole) of (I) and 53.6 mg (0.175 mmole) of (III) in 9 ml of methanol, and the other being 1 ml of 0.190 *M* methanol solution of hydrogen chloride. The solutions were degassed by several freeze-thawings *in vacuo*. The tube was then sealed with evacuation and the two solutions were mixed at room temperature. It was allowed to stand at room temperature. Yellow-orange crystals precipitated after standing for about 2 h. After standing for 75 h, the reaction mixture was dissolved in benzene (100 ml) and the solution was washed with a saturated sodium bicarbonate solution and water, and dried over anhydrous sodium sulfate. After evaporation of the solvent, the residue was chromatographed on alumina. Elution firstly with ether gave 63.4 mg (65.8% yield) of (V_a) and secondly with benzene gave 27.9 mg (29.0% yield) of (V_b).

It was confirmed that attempts to cause (I) or (III) to react alone under the same conditions as above failed, and only the starting substance was recovered unchanged.

(*b*). Similarly, (II) (201 mg, 0.555 mmole) and (IV) (144 mg, 0.450 mmole) were allowed to react in 0.112 *M* methanol solution (10 ml) of hydrogen chloride at room temperature for 168 h. After work-up as above, chromatography on alumina using benzene as an eluant gave 236 mg (90.7 % yield) of a mixture of two diastereomers of 1,2-diferrocenyl-1,2-di-*p*-tolylethane (VI). The mass spectrum of the mixture had peaks at 578 (M^+, 2.7), 289 (M^{2+} or $C_5H_5FeC_5H_4CHC_6H_4Me^+$, 100), 167 ($C_6H_4$-$C_6H_4Me+H^+$, 7.1), 141 (2.7), 121 ($C_5H_5Fe^+$, 28); and 56 ($Fe^+$, 3.0%). The mixture was repeatedly chromatographed on alumina. Elution firstly with ether gave 148 mg (56.9% yield) of (VI_a) and secondly with benzene gave 69.8 mg (26.8% yield) of (VI_b).

Attempts to bring (II) or (IV) separately into reaction under similar conditions failed, resulting only in recovery of the starting substance.

(*c*). Compounds (I) (146 mg, 0.419 mmole) and (IV) (134 mg, 0.419 mmole) were allowed to react as above in 0.112 *M* methanol solution (10 ml) of hydrogen chloride at room temperature for 137 h. After work-up, chromatography on alumina using benzene as an eluant gave 230 mg of a mixture of three coupling products (V), (VI), and 1,2-diferrocenyl-1-phenyl-2-*p*-tolylethane (VII), m.p. 177–184°. The NMR spectrum of the mixture exhibited absorptions at 7.73 [bs, CH_3 of (VII)], 7.65 [bs, CH_3 of (VI)], 6.40 (s, C_5H_5), 6.37 (s, C_5H_5), 6.18 (bs, C_5H_4), 3.00 (bs, C_6H_4), and 2.85 ppm (bs, C_6H_5). Their relative intensities were 9/13/84/56/28/34. Mass spectrum: *m/e* 578 (3.4), 564 (9.0), 550 (3.7), 289 (100), 275 (55), 167 (4.3), 153 (5.0), 121 (34), and 56 (5.5%). The mixture was determined to consist of (V) (23%), (VI) (22%), and (VII) (55%) on the basis of the mass spectrum, or alternatively, (V) (25%), (VI) (31%), and (VII) (44%) by the NMR spectrum.

(*d*). Compounds (II) (194 mg, 0.536 mmole) and (III) (164 mg, 0.536 mmole) were allowed to react in 0.112 *M* methanol solution (10 ml) of hydrogen chloride at room temperature for 137 h. After work-up, chromatography on alumina using benzene as an eluant yielded 295 mg of a mixture of (V), (VI), and (VII), m.p. 170–175°. NMR: 7.75 [bs, CH_3 of (VII)], 7.67 (bs, CH_3 of (VI)], 6.42 (s, C_5H_5), 6.38 (s, C_5H_5), 6.21 (bs, C_5H_4), 3.06 (bs, C_6H_4), and 2.94 ppm (bs, C_6H_5). Their relative intensities are 3/5/34/24/10/11. Mass spectrum: *m/e* 578 (2.2), 564 (6.9), 550 (2.8), 289 (100), 275 (64), 167 (10), 153 (11), 121 (73) and 56 (9.5%). Therefore, the mixture was calculated to consist of (V) (23%), (VI) (19%), and (VII) (58%) by the mass spectrum, or alternatively, (V) (22%), (VI) (36%), and (VII) (43%) by the NMR spectrum.

Acid-catalyzed ethanolysis of (III) and (IV)

Compound (III) (106 mg, 0.347 mmole) was allowed to react in an oxygen-free 0.042 *M* ethanol solution (10 ml) of hydrogen chloride at room temperature for 42 h. After work-up, chromatography on alumina using ether as an eluant gave 108 mg (97.3% yield) of α-ferrocenylbenzyl ethyl ether, m.p. 53–54° (lit.[10] m.p. 55–55.5°).

Similarly, (IV) (282 mg, 0.928 mmole) was allowed to react in 0.065 *M* ethanolic solution (20 ml) of hydrogen chloride at room temperature for 19 h. After work-up, chromatography on alumina using ether as an eluant gave 208 mg (57.8% yield) of oily *p*-methyl-α-ferrocenylbenzyl ethyl ether (Found: C, 71.75: H, 6.73. $C_{20}H_{22}FeO$ calcd.: C, 71.87: H, 6.63%). NMR: 8.80 (t, *J* 6.8 Hz, $OCCH_3$), 7.65 (s, *p*-CH_3), 6.60 (q, *J* 6.8 Hz, OCH_2), 6.00 (s, C_5H_5 and C_5H_4), 5.03 (s, CH), and 2.86 ppm ($A'_2B'_2$, C_6H_4).

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