REACTIVITY OF [*m*]FERROCENOPHANONES: THE ALDOL CONDENSATION

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Investigation of the reaction of [*m*]ferrocenophanones with *p*-chlorobenzaldehyde in basic medium showed that these cyclic ketones are much more reactive than their acyclic counterparts. The size of the bridge and the position of the carbonyl group influenced the reaction. Thus, [*m*]ferrocenophan-1-ones ($m = 3, 4$) afforded β-hydroxyketones only, [5]ferrocenophan-1-one gave in addition an α,β-unsaturated ketone, and [4]ferrocenophane-2-one yielded only α,β-unsaturated ketones. Oxidation of [*m*]ferrocenophanes with MnO_2 furnished the expected monoketones and [4]ferrocenophane-1,4-dione and [5]ferrocenophane-1,2-dione. The preparation of [5]ferrocenophane-1,5-dione was also improved.

Apart from preparation of [*m*]ferrocenophanes and [*m*]ferrocenophanones[1-4] no considerable attention has been paid to their reactions at the bridge carbon atoms. Although the aldol condensation has frequently been employed in the organic synthesis[5,6], no suitable conditions have been found for reactions of aldehydes with higher aliphatic ketones excepting alkyl methyl ketones[5] and 3-pentanone[7]. This fact can be rationalized by the reverse reaction, *i.e.* decomposition of the β-hydroxyketone is much faster than the elimination of water leading to α,β-unsaturated ketones[8]. Also the reaction of alkyl aryl ketones with aromatic aldehydes proceeds in basic medium with low yields[9] the exception being phenacylbenzene, which reacted with *p*-nitrobenzaldehyde to give stereoisomeric β-hydroxyketones in high yield[10]; this could be, however, due to their low solubility in the reaction medium. On the other hand, 1-indanone[11] and tetralone[12,13] react quite well in basic medium with aromatic aldehydes, whilst cyclohexanone afforded, according to reaction conditions either a product of one-side[14], or symmetric[15] condensation.

This paper is aimed to investigate the reactivity of [*m*]ferrocenophanones with *p*-chlorobenzaldehyde under conditions of a base catalyzed reaction and to compare the reactivity of these cyclic alkyl aryl ketones with the selected alkyl ferrocenyl ketones.

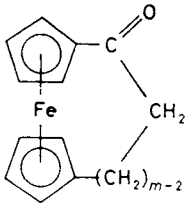
The starting [*m*]ferrocenophanones were prepared according to procedures already described, the exception being an improvement of the synthesis of [5]ferro-

cenophane-1,5-dione: the mixture of products obtained upon acylation of acetylferrocene with 3-chloropropionyl chloride was not separated, but transferred into an alkaline medium, where elimination of hydrogen chloride and cyclization of the originated 1-acetyl-1'-acryloylferrocene took place to yield [5]ferrocenophane-1,5--dione. [*m*]Ferrocenophan-1-ones (m = 4,5) could be prepared[16] by oxidation of [*m*]ferrocenophanes (m = 4,5) with active MnO_2. Manganese dioxide prepared for this purpose from $MnCl_2$ and $KMnO_4$ according to[17] was so active that [4]ferrocenophane gave, in addition to the expected [4]ferrocenophan-1-one (40%) also [4]ferrocenophane-1,4-dione in a 15% yield. An analogous oxidation of [5]ferrocenophane afforded, besides of [5]ferrocenophan-1-one, a hitherto not described [5]ferrocenophane-1,2-dione in a 3·7% yield.

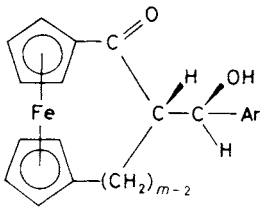
Reactions of [*m*]ferrocenophan-1-ones with *p*-chlorobenzaldehyde were carried out under conditions described for condensation of acetylferrocene[17] with various aromatic aldehydes with high yields. The presented data show that no reaction under investigation lead to results similar to those of reaction of tetralone with benzaldehyde[12,13], where the 2-benzylidene derivative was obtained in a 88% yield. However, the reaction course considerably depends on the size of the bridge in the respective [*m*]ferrocenophan-1-one. First of all, the overal yields of the reaction descreased with the increasing length of the bridge (with the increasing m); further, compounds with m = 3, 4 afforded only stereoisomeric β-hydroxyketones (the assignment is random), compound with m = 5 gave also an unsaturated ketone. Explanation lies in the structure of the starting [*m*]ferrocenophan-1-ones, the rigidity of which decreases from m = 3 to m = 5. The strain of [*m*]ferrocenophanes is compensated by a ring-tilting effect, or by a deviation of the carbonyl group out of the coplanarity with the cyclopentadienyl ring. This deviation rises with the decreasing m, or with the increasing number of carbons forming the bridge which are in an sp^2 hybridization. It has been found[19] that the ring tilting of [4]ferrocenophan-1-one (*Ib*) was 4·4°, and deviation of —CO— from coplanarity 18·3°; with [3]ferrocenophan-1-one[20] (*Ia*) these values were 8·8 and 42°, respectively, and with [3]ferrocenophane-1,3-dione[21] up to 9·8 and 38·6 or 45·2°, respectively. The increasing strain in the coumpoud is associated with a decrease of thermodynamic stability, what was reflected by an easy opening of the [3]ferrocenophane-1,3-dione ring[22], or by an extension of the 2-benzylidene [3]ferrocenophane-1,3-dione ring[23].

The presented data reveal that the carbonyl group of [3]ferrocenophan-1-one (*Ia*) is mostly deviated from coplanarity with the cyclopentadienyl ring. The resonance effect could not come too much into effect and therefore, the acidity of hydrogens of the neighbouring group rises. This fact could be responsible for the highest yields of the β-hydroxyketone. The elimination of water does not take place, since further sp^2 hybridized carbon would enter the bridge, and consequently, the strain of the system would increase. Since [5]ferrocenophan-1-one (*Ic*) does practically not reveal a strain also an unsaturated ketone was formed. Attempts to react [3]ferro-

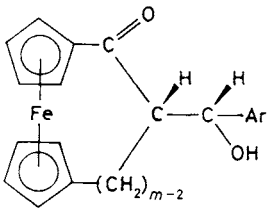
cenophan-1-one (*Ia*) with *p*-chlorobenzaldehyde under catalysis of piperidine or tert-potassium butoxide (a not separable mixture of 5 compounds), or to dehydrate 2-((4-chlorophenyl)hydroxymethyl)[3]ferrocenophan-1-one (*IIa*) with *p*-toluenesulfonic acid failed.



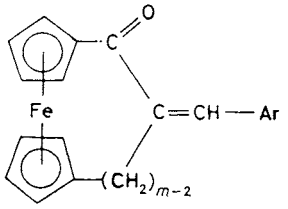
Ia – Ic



II a–c

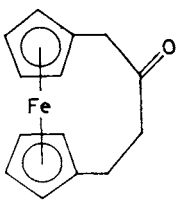


III b–c

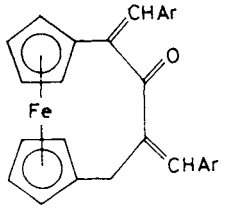


IV c

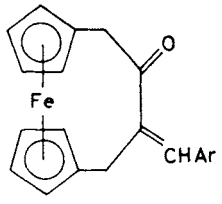
I *Vc*, Ar = *p*-ClC_6H_4; *a*, *m* = 3; *b*, *m* = 4; *c*, *m* = 5.



V



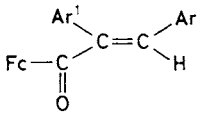
VI, Ar = *p*-ClC_6H_4



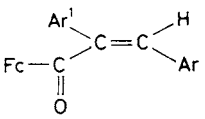
VII, Ar = *p*-ClC_6H_4

An attempt to react the analogous [4]ferrocenophan-2-one (*V*) showed that this derivative resembled simple cyclic ketones[14,15]. Since the carbonyl group is isolated from the cyclopentadienyl rings by methylene groups, the starting material does not have an internal strain and therefore, the plane of the enone in the reaction products would be almost perpendicular to the cyclopentadiene ring plane similarly as with 2-benzylidene[3]ferrocenophane-1,3-dione[24].

Condensations of alkyl ferrocenyl ketones with *p*-chlorobenzaldehyde under catalysis of potassium hydroxide and tert-potassium butoxide were performed for comparison purpose. An attempt to react propionylferrocene failed. A small amout of two and six compounds, respectively, was obtained, the products have not been isolated. The failure could be rationalized by a rapid decomposition of the β-hydroxyketone formed. An independent experiment showed that the corresponding chalcone (prepared by acylation of ferrocene with α-methylcinnamoyl chloride) is stable under these reaction conditions. The reaction of phenylacetylferrocene gave under the afore-mentioned conditions a small amount of one, and in the second case of both possible unsaturated ketones.



VIII



IX

VIII, *IX* Ar = *p*−ClC_6H_4, $Ar^1 = C_6H_5$, Fc = ferrocenyl

EXPERIMENTAL

The starting ferrocenes were prepared as follows: [3]ferrocenophan-1-one (*Ia*) according to[25], [4]ferrocenophan-2-one (*V*) according to[26], [5]ferrocenophane according to[16], propionylferrocene and phenylacetylferrocene according to[27,28], respectively. Melting points of products were measured with a Kofler micro hot-stage, the ^1H NMR spectra were run with a Tesla BS 487 spectrometer operating at 80 MHz. The spectra were recorded in C^2HCl_3 (99·5% of ^2H-isotope) tetramethylsilane being the internal reference. The chemical shift values are given in ppm at the δ scale.

[5]Ferrocenophane-1,5-dione

To the stirred solution of acetylferrocene (13 g, 5·7 mmol) in dichloromethane (200 ml) $AlCl_3$ (11·3 g, 8·5 mmol) was added; a complex prepared by mixing 3-chloropropionyl chloride (10·9 g, 6 mmol) with $AlCl_3$ (11·3 g, 8·5 mmol) in dichloromethane (200 ml) was added during 2 h with cooling. The mixture was then stirred at room temperature for 4 h and poured into water. The organic material was taken with dichloromethane, the extract was well washed with water and dried. The solvent was removed. Yield 15·7 g of a red oil which was dissolved in ethanol (50 ml) and poured into a stirred solution of 10%-NaOH (150 ml) diluted with ethanol (15 ml). The mixture was refluxed for 1 h, ethanol was distilled off and the brown product precipitating after cooling was filtered off, washed with water and dried. The residue was extracted with light petroleum in a Soxhlet apparatus and the extract was worked up. Yield 2·9 g (18%) of acetylferrocene; 9·8 g (90%) of the brown product, m.p. 251–254°C (ref.[16] 251–253°C) left in the apparatus.

Oxidation of [4]Ferrocenophane with MnO_2

Manganese dioxide (10 g) prepared according to[17] was added to the benzene (200 ml) solution of [4]ferrocenophane (2 g, 8·3 mmol). The mixture was filtered after a 3-day standing at room temperature, the solvent was evaporated and the residue was chromatographed over alumina. The unreacted [4]ferrocenophane (0·4 g, 20%) was eluted with benzene, [4]ferrocenophan-1-one (*Ib*, 0·7 g, 33%) (its m.p. 98–101°C was in accordance with[26]) was eluted with benzene–ether, and [4]ferrocenophane-1,4-dione, m.p. 172–175°C (0·30 g, 13%) was eluted with ether. For $C_{14}H_{12}FeO_2$ (268·1) calculated: 62·74% C, 4·51% H, 20·84% Fe; found: 62·32% C, 4·57% H, 21·13% Fe. ^{1}H NMR spectrum: 4·81 (4 H, s, H_α), 4·55 (4 H, s, H_β), 2·96 (4 H, s, CH_4).

Oxidation of [5]Ferrocenophane with MnO_2

Manganese dioxide (25 g) prepared according to[17] was added to the solution of [5]ferrocenophane (4·8 g, 19 mmol) in benzene (30g ml) and the mixture was worked up as in the preceding case. Elutions with light petroleum gave the unreacted [5]ferrocenophane (1·4 g, 29%), with light petroleum–ether (1 : 1) [5]ferrocenophan-1-one (*Ic*) (1·5 g, 30%, m.p. 123–125°C) and with ether [5]ferrocenophane-1,2-dione (0·2 g, 3·7%) m.p. 138–139°C (acetone–ether). For $C_{15}H_{14}FeO_2$ (282·2) calculated: 63·87% C, 5·0% H, 19·80% Fe; found: 63·77% C, 5·09% H, 19·64% Fe. ^{1}H NMR spectrum: 4·88 (2 H, t, H_α), 4·70 (2 H, t, H_β), 4·08 (4 H, q, C_5H_4—H_α, H_β), 2·87 (2 H, m, $COCH_4$), 2·46 (2 H, m, C_5H_4—CH_2), 2·00 (2 H, m, CH_2).

Condensation of [*m*]Ferrocenophanones with *p*-Chlorobenzaldehyde

A solution of potassium hydroxide (0·08 g, 1·5 mmol) in water (1 ml) was added to a mixture of [*m*]ferrocenophanone (1·5 mmol) and *p*-chlorobenzaldehyde (0·2 g, 1·5 mmol) in ethanol (5 ml). The mixture was stirred at room temperature for 24 h, the precipitate was filtered off (the case of [3]ferrocenophan-1-one), the filtrate was poured into water, the organic material was taken out with dichloromethane, the solvent was dried (Na_2SO_4), evaporated and the residue was chromatographed on a silica gel column with benzene–ethyl acetate (19 : 1).

Condensation of [3]*ferrocenophan-1-one* (Ia): Filtration of the reaction mixture afforded 2-((4-chlorophenyl)hydroxymethyl)[3]ferrocenophan-1-one (*IIa*), (0·2g, 44%) m.p. 196–197°C (ethanol). For $C_{20}H_{17}ClFeO_4$ (380·6) calculated: 63·10% C, 4·5% H, 9·3% Cl; found: 62·68% C, 4·45% H, 9·18% Cl. ^{1}H NMR spectrum (CF_3COOH): 6·98 (4 H, s, Ar), 4·94 (1 H, m, Fc), 4·79 (1 H, d, J = 7 Hz, CH—Ar), 4·65 (1 H, m, Fc), 4·43 (4 H, m, Fc), 4·08 (1 H, m, CHCO), 3·73 (3 H, m, Fc, OH), 3·08 (1 H, dd, CH_2), 2·45 (1 H, dd, CH_4). Chromatography of the extract showed the presence of the product and the starting material in trace amounts.

Condensation of [4]*ferrocenophan-1-one* (Ib): Chromatography of the extract furnished the starting material (0·09 g, 24%) from the first chromatographic band, and *erythro*-2-((4-chlorophenyl)hydroxymethyl)[4]ferrocenophan-1-one (*IIb*) m.p. 172–174°C (ethanol) from the second band. For $C_{21}H_{19}ClFeO_2$ (394·7) calculated: 63·9% C, 4·85% H, 14·15% Fe; found: 61·93% C, 4·55% H, 14·81% Fe. ^{1}H NMR spectrum: 7·35 (4 H, s, Ar), 5·17 (1 H, d, J = 3·8 Hz, CH—Ar), 4·75 and 4·66 (1 H and 1 H, m, Fc—H_α), 4·55 (2 H, m, Fc—H_β), 4·21, 4·08 and 3·90 (1 H, 2 H and 1 H, m, Fc—$H_{\alpha',\beta'}$), 4·3 (1 H, bs, OH), 2·89 (1 H, m, CH), 2·16 (4 H, m, CH_2).

threo-2-((4-Chlorophenyl)hydroxymethyl)[4]ferrocenophan-1-one (*IIIb*) (0·05 g, 8·5%), m.p. 156–158°C (ethanol) was isolated from the last band. For $C_{21}H_{19}ClFeO_2$ (394·7) calculated: 63·9% C, 4·85% H, 14·15% Fe; found: 62·5% C, 4·90% H, 14·30% Fe. ^{1}H NMR spectrum: 7·31 (4 H, s, Ar), 4·94 (1 H, d, J = 6 Hz, CH—Ar), 4·65 (2 H, m, Fc—H_α), 4·49 (2 H, m, Fc—H_β), 4·20, 4·08 and 3·89 (1 H, 2 H and 1 H, m, Fc—$H_{\alpha',\beta'}$), 3·08 (1 H, m, CH), 2·20 (4 H, m, CH_2).

Condensation of [5]*ferrocenophan-1-one* (Ic): Chromatographic separation afforded 2-(4-chlorophenylmethylene)[5]ferrocenophan-1-one (*IVc*) 0·1 g (17%), m.p. 162–163°C (ethanol). For $C_{22}H_{19}ClFeO$ (390·7) calculated: 67·62% C, 4·90% H, 14·30% Fe; found: 66·78% C, 5·00% H, 14·05% Fe. ^{1}H NMR spectrum: 7·31 (4 H, s, Ar), 6·96 (1 H, s, CH), 4·73 (2 H, m, Fc—H_α), 4·58 (2 H, t, Fc—H_β), 4·18 (4 H, m, Fc—$H_{\alpha',\beta'}$), 3·16 (2 H, m, CH_2—C=), 2·43 (2 H, m, CH_2Fc), 1·60 (2 H, m, —CH_2—). The next band contained the starting material (0·2 g, 50%) and the third band 2-((4-chlorophenyl)hydroxymethyl)[5]ferrocenophan-1-one (*IIc*) 0·1 g, (8·2%), m.p. 170·5–171·5°C (ethanol). For $C_{22}H_{21}ClFeO_2$ (408·7) calculated: 64·64% C, 5·18% H, 13·65% Fe; found: 63·84 C, 5·27% H, 14·37% Fe. ^{1}H NMR spectrum: 7·30 (4 H, s, Ar), 4·95 (1 H, d, J = 4 Hz, CH—Ar), 4·83 (1 H, bs, OH), 4·55 (4 H, m, Fc), 4·08 (4 H, m, Fc), 3·08 (1 H, m, CH), 2·26 (2 H, m, CH_2), 1·83 (4 H, m, CH_2). The last band contained 2-((4-chlorophenyl)hydroxymethyl)[5]ferrocenophan-1-one (*IIIc*) 0·08 g, 6·5%, m.p. 136–137°C (ethanol). For $C_{22}H_{21}ClFeO_2$ (408·7) calculated: 64·64% C, 5·18% H, 13·6% Fe; found: 63·93% C, 5·49% H, 13·56% Fe. ^{1}H NMR spectrum: 7·29 (4 H, s, Ar), 4·79 (2 H, m, Fc, CH—Ar), 4·50 (3 H, m, Fc), 4·06 (4 H, m, Fc), 3·26 (2 H, m, CH, OH), 2·25 (2 H, m, CH_2), 1·65 (4 H, m, CH_2).

Condensation of [4]*ferrocenophan-2-one* (V): Chromatographic separation furnished 1,3-bis-(4-chlorophenylmethylene)[4]ferrocenophan-2-one (*VI*), 0·13 g (19·2%), m.p. 209–213°C (ethanol). For $C_{28}H_{20}Cl_2FeO$ (499·2) calculated: 67·36% C, 4·0% H, 11·18% Fe; found: 66·96% C, 4·01% H, 11·15% Fe. ^{1}H NMR spectrum: 7·84 (1 H, s, =CH—), 7·38 (4 H, dd, Ar), 7·25 (4 H, s, Ar), 6·44 (1 H, s, =CH—), 4·28 (2 H, t, Fc—H_α), 4·13 (6 H, m, Fc—$H_{\alpha'}$, H_β, $H_{\beta'}$), 3·60 (2 H, s, CH_2). The second band afforded 3-(4-chlorophenylmethylene)[4]ferrocenophan-2-one (*VII*), m.p. 208–212°C (ethanol). For $C_{21}H_{17}ClFeO$ (376·7) calculated: 66·95% C, 4·55% H, 14·82% Fe; found: 67·27% C, 4·58% H, 14·70% Fe. ^{1}H NMR spectrum: 7·77 (1 H, s, =CH), 7·34 (4 H, s, Ar), 4·1 (8 H, m, Fc), 3·61 (2 H, s, CH_2), 3·54 (2 H, s, CH_2). The last band yielded the starting product (0·03 g, 7·5%).

Condensation of Phenylacetylferrocene with *p*-Chlorobenzaldehyde

A) A solution of potassium hydroxide (0·16 g) in water (1 ml) was added to the mixture consisting of phenylacetylferrocene (1·1 g, 3·9 mmol) and 4-chlorobenzaldehyde (0·55 g, 3·3 mmol) in ethanol (10 ml). The mixture was stirred at room temperature for 24 h, poured into water, the organic material was extracted with dichloromethane, the extract was dried (Na_2SO_4), the solvent was distilled off and the residue was chromatographed over silica gel with benzene–ethyl acetate (19 : 1). Yield 0·13 g (8·6%) of (*Z*)-3-(4-chlorophenyl)-2-phenyl-1-ferrocenylpropenone (*IX*) m.p. 184–186°C (ethanol). For $C_{25}H_{19}ClFeO$ (426·7) calculated: 70·36% C, 4·48% H; found: 69·82% C, 4·38% H. ^{1}H NMR spectrum: 7·38 (5 H, Ar, =CH), 7·28 (2 H, m, Ar), 7·09 (3 H, m, Ar), 4·65 (2 H, t, Fc-H_α), 4·48 (2 H, t, Fc—H_β), 4·11 (5 H, s, C_5H_5). Starting material (0·9 g, 8%) was obtained from the next band.

B) A solution of phenylacetylferrocene (1·1 g, 3·9 mmol) and 4-chlorobenzaldehyde (0·55 g, 3·9 mmol) in toluene (20 ml) was added into tert-potassium butoxide (0·45 g, 3·9 mmol) in toluene (5 ml), the mixture was refluxed for 8 h, poured into water and the product was taken with dichloromethane. The extract was dried (Na_2SO_4), the solvent was evaporated and the residue was chromatographed on a silica gel-packed column with benzene–ethyl acetate (19 : 1). Yield 0·24 g (16%) of (*E*)-3-(4-chlorophenyl)-2-phenyl-1-ferrocenylpropenone (*VIII*), m.p. 192–194°C (ethanol). For $C_{25}H_{19}ClFeO$ (426·7) calculated: 70·36% C, 4·48% H; found: 69·58% C, 4·39% H. ^{1}H NMR spectrum: 7·74 (2 H, m, Ar), 7·40 (3 H, m, Ar), 7·25 (4 H, m, Ar), 6·83 (1 H, s, =CH), 4·68 (2 H, t, Fc—H_α), 4·41 (2 H, t, Fc—H_β), 3·83 (5 H, s, C_5H_5). The second band afforded *IX* (0·17 g, 12%), m.p. 184–186°C (ethanol), and the last one the starting material (0·3 g, 27%).

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