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Reaction of Monocyclic Ferrocenyl-4,5-dihydropyrazoles with β-Dicarbonyl Compounds

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Abstract — Monocyclic 3- and 5-ferrocenyl-4,5-dihydropyrazoles with a free NH group in the molecule react with acetylacetone to form the corresponding enaminocarbonyl compounds. The latter were isolated as a single isomer, presumably *E*. 3-Ferrocenyldihydropyrazoles and 5- ferrocenyl-3-(*p*-methoxyphenyl)-4,5-dihydropyrazole analogously react with acetoacetic ester. 5-Ferrocenyl-3-phenyl-, 3-(*p*-bromophenyl)-5-ferrocenyl, and 3,5-diferrocenyl-4,5-dihydropyrazoles react with acetoacetic ester to form acetoacetylpyrazolides.

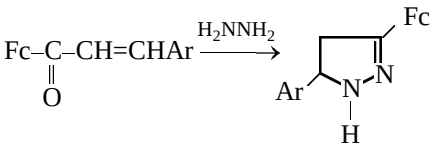
The great interest in ferrocenyl-substituted heterocyclic compounds is associated both with the peculiar chemical behavior of the ferrocene system and with the unusual properties it imparts to the heterocyclic fragment. Such compounds exhibit diverse biological activity and some other important properties [1–9].

Ferrocenyl-substituted 4,5-dihydropyrazoles are rather well studied [1–9]. They are prepared by addition of hydrazines to α,β-unsaturated carbonyl compounds. A series of stable dihydropyrazoles containing a ferrocenyl substituent and a substituent on N^1 are found to possess biological activity. In detail, 4-acetyl-3-ferrocenyl-1,4,5-triazatricyclo[5.2.2.$0^{2.6}$]-undec-5-ene exhibits a high antiviral activity [9]. Introduction in ferrocenyl-4,5-dihydropyrazole of additional carbo- and heterocyclic fragments, as well as various substituents on the N^1 atom of the pyrazole ring permits one to expect a broader spectrum of useful properties in the resulting products.

In the present work we have studied the reaction of monocyclic ferrocenyldihydropyrazoles with β-dicarbonyl compounds, such as acetylacetone and acetoacetic ester. Starting dihydropyrazoles **Ia**–**Ic** and **IIa**–**IId** were prepared by a standard procedure from corresponding α,β-unsaturated ketones **IIIa**–**IIIc** and **IVa**–**IVd** and hydrazine hydrate [4, 6, 9] (Scheme 1).

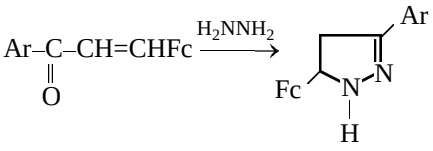
It was found that ferrocenyl-4,5-dihydropyrazoles **Ia**–**Ic** and **IIa**–**IId** react with acetylacetone under short reflux (about 5 min) to give enaminocarbonyl

Scheme 1.



IIIa–**IIIc** **Ia**–**Ic**

Ar = Ph (**a**), *p*-$CH_3OC_6H_4$ (**b**), *p*-BrC_6H_4 (**c**); Fc = $C_5H_5FeC_5H_4$.



IVa–**IVd** **IIa**–**IId**

Ar = Ph (**a**), *p*-$CH_3OC_6H_4$ (**b**), *p*-BrC_6H_4 (**c**), Fc (**d**).

derivatives **Va**–**Vc** and **VIa**–**VId**, respectively (Scheme 2).

Conclusive evidence for the structure of the resulting compounds was obtained from their ^1H and ^{13}C NMR spectra and elemental analyses (see Experimental).

The ^1H NMR spectra of products **Va**–**Vc** and **VIa**–**VId** contain an *ABX* proton system characteristic of dihydropyrazoles and vary significantly depending on the position of the ferrocenyl substituent in the heteroring. In 3-ferrocenyldihydropyrazoles **Va**–**Vc**, the

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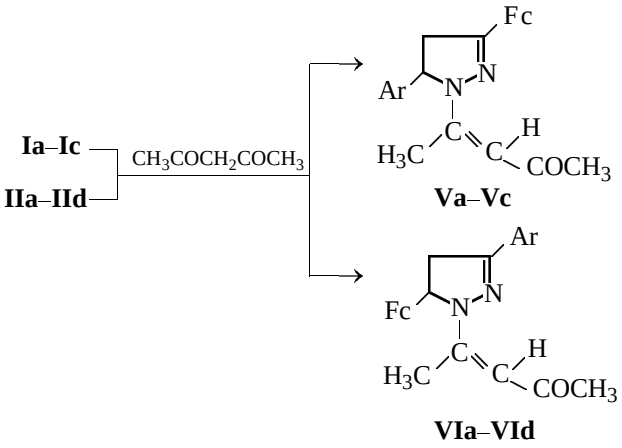
Scheme 2.



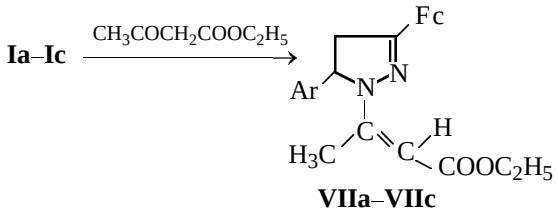
geminal H_A and H_B proton signals appear at δ 2.88–3.71 ppm (Δδ 0.74–0.82 ppm). Therewith, the olefin proton signals of the substituent on N^1 have a slightly different chemical shift than those of H_X (δ ~5.14–5.18 and 5.20–5.24 ppm). Contrary to that, in 5-ferrocenyldihydropyrazoles **VIa**–**c**, one of the methylene proton signals is shifted noticeably downfield (δ ~3.65–3.70 ppm), Δδ ~0.01–0.09 ppm, and the olefin proton signals (δ ~5.46 ppm) are located downfield from the heteroring H_X proton signals (δ ~5.20 ppm). In the presence of two ferrocenyl substituents in the 3 and 5 positions of the heteroring (compound **VId**), the chemical shifts of heteroring and olefin protons are close to those in 5-ferrocenyl derivatives **VIa**–**VIc**.

Position of the ferrocenyl group in 1-(1-methyl-3-oxopent-2-en-1-yl)-4,5-dihydropyrazoles influences chemical shifts of its protons. Hence, in 3-ferrocenyl derivatives **Va**–**Vc**, all proton signals of the substituted ferrocene cyclopentadienyl ring are located downfield from the signal of the unsubstituted cyclopentadienyl ring. In 5-ferrocenyldihydropyrazoles **VIa**–**VIc** containing the ferrocenyl substituent on an sp^3 carbon atom, part of the signals of the substituted cyclopentadienyl ring are located upfield from the C_5H_5 proton signal.

Further evidence for the structure of enaminocarbonyl dihydropyrazole derivatives **Va**–**Vc** and **VIa**–**VId** was provided by the observation in the ^{13}C NMR spectra of expected number of signals of quaternary, as well as methyl, methylene, methine, aryl, and ferrocenyl carbon atoms. A significant specific feature of the ^{13}C NMR spectra of compounds **Va**–**Vc** is that the $C_{ipso}Fc$ carbon signals are observed upfield (δ ~75.0 ppm) compared with the respective signals of dihydropyrazoles **VIa**–**VIc** (δ ~89.0 ppm).

Further we found that pyrazolines **Ia**–**Ic** analogously react with acetoacetic ester to give compounds **VIIa**–**VIIc** (Scheme 3).

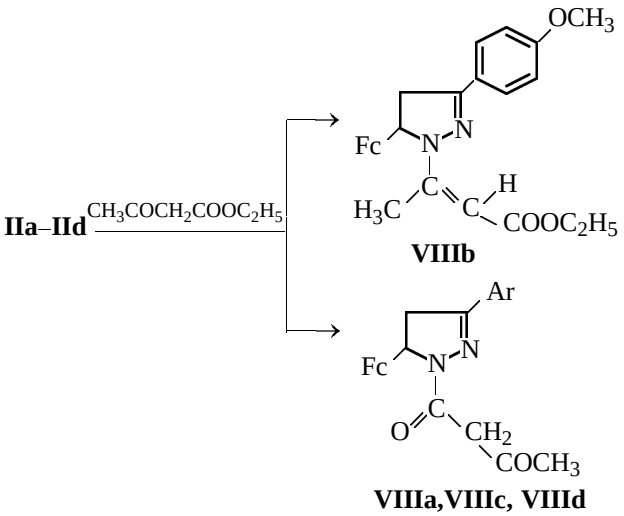
Scheme 3.



The structure of products **VIIa**–**VIIc** was proved by means of ^1H and ^{13}C NMR spectra (Tables 1 and 2) which contain well-defined signals of the *ABX* system of the dihydropyrazoline ring, a triplet and a singlet of two methyl groups, as well as a singlet of the olefin proton. The ^1H and ^{13}C NMR spectra of compounds **VIIa**–**VIIc** have common characteristics with those of compounds **Va**–**Vc**, but the olefin proton singlet of dihydropyrazoles **VIIa**–**VIIc** is located upfield (δ ~4.40–4.70 ppm) from the respective signal of compounds **Va**–**Vc**.

Unlike what is observed with dihydropyrazoles **Ia**–**Ic**, compound **IIb** is the only of pyrazolines **IIa**–**IId**, whose reaction with acetoacetic ester provides an enaminocarbonyl derivative (compound **VIIIb**). The other 5-ferrocenyl-4,5-dihydropyrazolines **IIa**, **IIc**, and **IId** react with acetoacetic acid under a more prolonged heating to give acetoacetic pyrazolides **VIIIa, VIIIc**, and **VIIId**, respectively (Scheme 4).

Scheme 4.



The ^1H NMR spectra of pyrazolides **VIIIa, VIIIc**, and **VIIId** have preserved the pyrazoline *ABX* proton system. They lack the singlet of the olefin proton and the triplet of the ethoxyethyl methyl protons and

Table 1. ^{1}H NMR spectra of compounds **IIa, IIc, IId, Va–Vc, VIa–VId, VIIa–VIIc**, and **VIIIa–VIIId**, δ, ppm (*J*, Hz)

Comp. no.	C_5H_5, s	C_5H_4, m	CH_3, CH_2	CH, NH	Ar
IIa	4.20 (5H)	4.13 (4H)	3.05 d.d (1H, *J* 5.4, 16.2), 3.33 d.d (1H, *J* 10.2, 16.2)	4.66 d.d (1H, *J* 5.4, 10.2), 5.84 br.s (1H)	6.92 d (2H, *J* 8.7), 7.49 d (2H, *J* 8.7)
IIc	4.20 (5H)	4.14 (4H)	3.00 d.d (1H, *J* 5.7, 16.2), 3.31 d.d (1H, *J* 10.5, 16.2)	4.67 d.d (1H, *J* 5.7, 10.5), 5.87 br.s (1H)	7.48 d (2H, *J* 9.0), 7.52 d (2H, *J* 9.0)
IId	4.13 (5H) 4.21 (5H)	4.16 (4H), 4.31 (2H), 4.56 (2H)	2.84 d.d (1H, *J* 3.9, 15.9), 3.22 d.d (1H, *J* 10.5, 15.9)	4.52 d.d (1H, *J* 3.9, 10.5), 5.61 br.s (1H)	–
Va	4.07 (5H)	4.39 (2H), 4.50 (1H), 4.68 (1H)	1.97 s (3H), 2.67 s (3H), 2.96 d.d (1H, *J* 3.9, 17.1), 3.71 d.d (1H, *J* 11.4, 17.1)	5.18 br.s (1H), 5.20 d.d (1H, *J* 3.9, 11.4)	7.15–7.42 (5H)
Vb	4.08 (5H)	4.38 (2H), 4.50 (1H), 4.68 (1H)	1.98 s (3H), 2.66 s (3H), 2.94 d.d (1H, *J* 3.6, 17.1), 3.68 d.d (1H, *J* 11.4, 17.1), 3.79 s (3H)	5.21 br.s (1H), 5.23 d.d (1H, *J* 3.6, 11.4)	6.90 d (2H, 9.0), 7.11 d (2H, *J* 9.0)
Vc	4.08 (5H)	4.40 (2H), 4.52 (1H), 4.65 (1H)	1.98 s (3H), 2.67 s (3H), 2.92 d.d (1H, *J* 3.9, 17.1), 3.72 d.d (1H, *J* 11.4, 17.1)	5.15 br.s (1H), 5.22 d.d (1H, *J* 3.9, 11.4)	7.07 d (2H, *J* 8.2), 7.51 d (2H, *J* 8.2)
VIa	4.12 (5H)	4.08 (1H), 4.17 (1H), 4.21 (2H)	2.10 s (3H), 2.72 s (3H), 3.75 d.d (1H, *J* 6.3, 17.1), 3.78 d.d (1H, *J* 8.4, 17.1)	5.11 d.d (1H, *J* 6.3, 8.4), 5.46 s (1H)	7.43 m (3H), 7.83 m (2H) *J* 8.2)
VIb	4.12 (5H)	4.08 (1H), 4.16 (1H), 4.20 (2H)	2.09 s (3H), 2.71 s (3H), 3.72 d.d (1H, *J* 6.0, 11.4), 3.76 d.d (1H, *J* 8.6, 11.4), 3.78 s (3H)	5.09 d.d (1H, *J* 6.0, 8.6), 5.43 s (1H)	6.97 d (2H, *J* 9.0), 7.76 d (2H, *J* 9.0)
VIc	4.12 (5H)	4.06 (1H), 4.18 (1H), 4.21 (2H)	2.10 s (3H), 2.70 s (3H), 3.70 d.d (1H, *J* 5.4, 17.1), 3.74 d.d (1H, *J* 9.6, 17.1)	5.12 d.d (1H, *J* 5.4, 9.6), 5.46 s (1H)	7.57 d (2H, *J* 9.0), 7.67 d (2H, *J* 9.0)
VId	4.16 (5H), 4.21 (5H)	4.08 (1H), 4.23 (3H), 4.44 (2H), 4.61 (1H), 4.74 (1H)	2.10 s (3H), 2.66 s (3H), 3.49 d.d (1H, *J* 6.9, 16.8), 3.67 d.d (1H, *J* 10.5, 16.8)	5.06 d.d (1H, *J* 6.9, 10.5), 5.44 s (1H)	–
VIIa	4.05 (5H)	4.36 (2H), 4.47 (1H), 4.65 (1H)	1.20 t (3H, *J* 6.7), 2.64 s (3H), 2.91 d.d (1H, *J* 3.9, 16.5), 3.68 d.d (1H, *J* 11.7, 16.5), 4.00 q (2H, *J* 6.7)	4.77 br.s (1H), 5.24 d.d (1H, *J* 3.9, 11.7)	7.17–7.36 m (5H)
VIIb	4.07 (5H)	4.36 (2H), 4.48 (1H), 4.65 (1H)	1.21 t (3H, *J* 6.9), 2.63 s (3H), 2.90 d.d (1H, *J* 3.8, 17.0), 3.67 d.d (1H, *J* 11.4, 17.0), 3.78 s (3H), 4.01 q (2H, *J* 6.9)	4.77 br.s (1H), 5.20 d.d (1H, *J* 3.8, 11.4)	6.88 d (2H, *J* 8.7), 7.11 d (2H, *J* 8.7)
VIIc	4.07 (5H)	4.37 (2H), 4.49 (1H), 4.63 (1H)	1.21 t (3H, *J* 7.2), 2.64 s (3H), 2.88 d.d (1H, *J* 3.9, 17.1), 3.70 d.d (1H, *J* 12.0, 17.1), 4.02 q (2H, *J* 7.2)	4.67 br.s (1H), 5.19 d.d (1H, *J* 3.9, 12.0)	7.07 d (2H, *J* 8.4), 7.50 d *J* 8.4)
VIIIa	4.15 (5H)	4.03 (1H), 4.13 (1H), 4.18 (1H), 4.48 (1H)	2.21 s (3H), 3.56 d.d (1H, *J* 4.2, 17.4), 3.72 d.d (1H, *J* 10.8, 17.4), 3.75 d (1H, *J* 16.9), 3.85 d (1H, *J* 16.9)	5.52 d.d (1H, *J* 4.2, 10.8)	7.47 m (3H), 7.76 m (2H)
VIIIb	4.12 (5H)	4.07 (1H), 4.09 (1H), 4.14 (1H), 4.22 (1H)	1.25 t (3H, *J* 6.9), 2.67 s (3H), 3.65 d.d (1H, *J* 4.5, 16.8), 3.74 d.d (1H, *J* 10.4, 16.8), 3.86 s (3H), 4.18 q (2H, *J* 6.9)	4.98 s (1H), 5.06 d.d (1H, *J* 4.5, 10.4)	6.97 d (2H, *J* 8.4), 7.73 d (2H, *J* 8.4)

Table 1. (Contd.)

Comp. no.	C_5H_5, s	C_5H_4, m	CH_3, CH_2	CH, NH	Ar
VIIIc	4.14 (5H)	4.01 (1H), 4.13 (1H), 4.18 (1H), 4.47 (1H)	2.21 s (3H), 3.52 d.d (1H, *J* 3.9, 17.4), 3.69 d.d (1H, *J* 11.1, 17.4), 3.75 d (1H, *J* 15.9), 3.86 d (1H, *J* 15.9)	5.53 d.d (1H, *J* 3.9, 11.1)	7.57 d (2H, *J* 9.0), 7.63 d (2H, *J* 9.0)
VIIId	4.18 (5H), 4.21 (5H)	4.02 (1H), 4.15 (1H), 4.23 (2H), 4.45 (2H), 4.49 (1H), 4.68 (1H)	2.37 s (3H), 3.36 d.d (1H, *J* 3.9, 17.1), 3.62 d.d (1H, *J* 11.1, 17.1), 3.64 d (1H, *J* 15.6), 3.86 d (1H, *J* 15.6)	5.47 d.d (1H, *J* 3.9, 11.1)	–

Table 2. ^{13}C NMR spectra of compounds **Va**–**Vc, VIa**–**VId, VIIa**–**VIIc, VIIIa**, and **VIIIb** (75 MHz), δ, ppm

Comp. no.	C_5H_5	C_5H_4	$C_{ipso}Fc$	CH_2, CH_3	C	CH=	CH	C=O	Ar
Va	68.3	67.0, 67.6, 70.3, 70.5	75.5	16.3, 31.8, 44.5	141.0, 154.6, 155.0	96.8	62.1	195.5	124.9, 128.0, 129.2
Vb	69.3	67.0, 67.5, 70.3, 70.4	75.6	16.3, 31.8, 44.6, 55.3	133.1, 154.6, 155.1, 159.2	96.7	61.7	195.5	114.6, 126.2
Vc	69.3	67.0, 67.5, 70.4, 70.5	75.2	16.3, 31.8, 44.4 154.4	121.8, 140.0,	96.9	61.5	195.5	126.7, 132.4
VIa	68.7	66.6, 67.8, 68.3, 68.4	88.5	16.4, 32.0, 41.4 155.8	131.6, 152.7,	97.6	57.7	195.5	126.3, 128.8, 129.9
VIb	68.5	66.5, 67.8, 67.9, 68.8	91.7	16.8, 31.8, 46.2, 55.5	118.8, 129.9, 145.7, 163.7	100.4	65.9	191.1	113.7, 130.5
VIc	68.8	66.5, 67.9, 68.4, 68.5	88.4	19.0, 20.5, 23.4, 24.2	124.2, 130.6, 151.4, 155.6	98.1	57.9	195.7	127.7, 132.1
VId	68.8, 69.5	66.2, 67.2, 67.6, 67.7, 68.4, 68.7, 70.2, 70.5	75.7, 88.8	16.6, 31.9, 43.4	155.0, 155.7	96.4	57.2	195.2	–
VIIa	69.2	67.4, 68.8, 70.0, 70.2	75.9	14.6, 15.9, 44.6, 55.3, 58.4	141.2, 152.8, 155.4	86.5	62.1	168.9	125.0, 127.8, 129.2
VIIb	69.3	66.8, 67.4, 70.0, 70.2	76.1	14.6, 15.9, 44.7, 55.3, 58.5	133.4, 152.8, 155.5, 159.1	86.4	61.6	169.0	114.5, 126.2
VIIc	69.3	66.9, 67.3, 70.2, 70.3	75.7	14.6, 15.9, 44.5, 58.6	121.7, 140.2, 152.7, 155.3	86.8	61.5	168.8	126.9, 132.4
VIIIa	68.6	65.5, 68.3, 68.4, 70.1	86.8	30.1, 39.9, 50.7	131.1, 155.0	–	55.6	164.8, 202.0	126.6, 128.8, 130.3
VIIIb	68.7	66.6, 67.8, 68.1, 68.4	88.8	14.6, 16.0, 42.0, 55.4, 57.5	124.5, 151.2, 156.2, 160.9	86.9	58.5	168.9	114.2, 127.7

contain a singlet of methyl, two doublets of methylene, and expected numbers of ferrocenyl and aryl proton signals.

After short refluxing (~5 min) of pyrazolines **IIa, IIc**, and **IId** with acetoacetic ester, starting compounds **IIa, IIb**, and **IId** were isolated and characterized by ^1H NMR spectra (Table 1).

Note that the reaction of ferrocenyl-4,5-dihydro-

Table 3. Yields, melting points, and elemental analyses of compounds **IIa, IIc, IId, Va**–**Vc, VIa**–**VId, VIIa**–**VIIc**, and **VIIIa**–**VIIId**

Comp. no.	Yield, %	mp, °C	Found, %				Formula	Calculated, %			
			C	H	Fe	N		C	H	Fe	N
IIa	59	113–114	69.23	5.41	16.86	8.25	$C_{19}H_{18}FeN_2$	69.11	5.50	16.91	8.48
IIc	61	134–135	55.64	4.28	13.81	6.77	$C_{19}H_{17}BrFeN_2$	55.78	4.19	13.65	6.85
IId	57	164–165	63.19	4.92	25.67	6.57	$C_{23}H_{22}Fe_2N_2$	63.05	5.06	25.50	6.39
Va	68	183–184	69.75	6.11	13.67	6.67	$C_{24}H_{24}FeN_2O$	69.91	5.87	13.55	6.79
Vb	70	172–173	67.69	5.74	12.91	6.21	$C_{25}H_{26}FeN_2O_2$	67.88	5.92	12.63	6.33
Vc	71	211–213	58.83	4.92	11.18	5.83	$C_{24}H_{23}BrFeN_2O$	58.68	4.72	11.37	5.70
VIa	69	173–174	70.11	5.69	13.42	6.97	$C_{24}H_{24}FeN_2O$	69.91	5.87	13.55	6.79
VIb	72	164–165	67.74	6.16	12.48	6.47	$C_{25}H_{26}FeN_2O_2$	67.88	5.92	12.63	6.33
VIc	77	192–194	58.44	4.63	11.24	5.54	$C_{24}H_{23}BrFeN_2O$	58.68	4.72	11.37	5.70
VId	71	232–233	64.53	5.61	21.67	5.49	$C_{28}H_{28}Fe_2N_2O$	64.65	5.42	21.47	5.38
VIe	76	313–314	66.51	5.52	13.58	6.53	$C_{23}H_{22}FeN_2O_2$	66.68	5.35	13.49	6.76
VIIa	62	174–175	68.02	6.01	12.42	6.21	$C_{25}H_{26}FeN_2O_2$	67.88	5.92	12.63	6.33
VIIb	71	168–169	66.27	6.05	11.68	5.72	$C_{26}H_{28}FeN_2O_3$	66.11	5.98	11.82	5.93
VIIc	74	195–197	57.48	4.67	10.90	5.46	$C_{25}H_{25}BrFeN_2O_2$	57.61	4.83	10.72	5.37
VIIIa	63	187–188	66.49	5.51	13.64	6.67	$C_{23}H_{22}FeN_2O_2$	66.11	5.98	11.82	5.93
VIIIb	67	206–208	66.26	5.83	11.69	5.69	$C_{26}H_{28}FeN_2O_3$	64.90	5.06	21.55	5.40
VIIIc	68	202–203	55.89	4.38	11.47	5.80	$C_{23}H_{21}BrFeN_2O_2$	56.01	4.29	11.33	5.67
VIIId	59	224–226	62.27	4.84	21.28	5.42	$C_{27}H_{26}Fe_2N_2O_2$	62.10	5.02	21.40	5.36

pyrazoles with β-dicarbonyl compounds proceeds stereoselectively. In all the cases, enaminocarbonyl derivatives **Va**–**Vc, VIa**–**Vd, VIIa**–**Vc**, and **VIIIb** were obtained as a single, probably *E*, geometric isomer [10].

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were measured on a Varian Unity-Inova spectrometer (300 and 75 MHz, respectively) for $CDCl_3$ solutions against internal TMS. The ^1H and ^{13}C NMR spectral data are listed in Tables 1 and 2. Column chromatography was carried out on Al_2O_3 (Brockmann activity grade III), eluent hexane–ether (2 : 1 v/v). The yields and elemental analyses of the obtained compounds are listed in Table 3.

Reagents purchased from Aldrich were used: ferrocenecarbaldehyde, 99%; 4-methoxybenzaldehyde, 98%; acetylferrocene, 95%; 4-bromobenzaldehyde, 99%; 4-methoxyacetophenone, 99%; 4-bromoacetophenone, 98%; benzaldehyde, 99%; and acetophenone, 99%.

α,β-Unsaturated ketones **IIIa**–**IIIc** and **IVa**–**IVd** were prepared from acetylferrocene and corresponding carbaldehydes or ferrocenecarbaldehyde, and corresponding ketones in aqueous alcoholic alkali [5].

(*E*)-1-[2-(4-oxopent-2-en-1-yl)]-4,5-dihydropyrazoles Va–**Vc and VIa**–**VId**. A mixture of 3.3 mmol of dihydropyrazole **Ia**–**Ic, IIa**–**IId** and 5 ml of acetylacetone was refluxed for 5 min. After cooling, the reaction mixture was diluted with 50 ml of ether. Crystals formed and were filtered off, washed with ether, and dried in air. Purification was carried out by column chromatography.

Reaction of ferrocenyl-4,5-dihydropyrazoles Ia–**Ic and IIa**–**IId with acetoacetic ester** was carried out in a similar way. Reaction products were precipitated with ether and crystallized from benzene to obtain (*E*)-1-[2-(ethoxycarbonyl)-1-methylvinyl]-4,5-dihydropyrazoles **VIIa**–**VIIc** and **VIIIb** and starting pyrazoles **IIa, IIc**, and **IId** (Tables 1–3).

Synthesis of acetoacetic pyrazolides VIIIa, VIIIc, and VIIId. A mixture of 3.3 mmol of dihydropyrazole **IIa, IIc**, or **IId** and 5 ml of acetoacetic ether was refluxed for 30 min. Excess acetoacetic ester was removed by steam distillation, and the residue was subjected to column chromatography.

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