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The Synthesis of Ferrocenyl- and Ferrocenoylpyrimidines

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Abstract—New derivatives of pyrimidine were synthesized from ferrocenyl ketones by the reactions of [3+1+1+1]annulation and intermolecular cyclization. The electrochemical behavior of the obtained compounds was studied by the method of cyclic voltammetry. All the compounds may be characterized with the signal, corresponding to the reversible one-electron transfer in ferrocene-ferrecinium and lowering of the redox potential with respect to ferrocene.

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Pyrimidine is one of the most wide spread natural heterocycles and it is a key fragment of many bioactive substances (like DNA, thiamine) and pharmaceuticals (luminal). The fragments of 4,5-disubstitued pyrimidine are often found in naturally occurring bioactive agents (vorikonazol [1] and avitriptan [2]).

The specific properties of ferrocene, including its high stability, capability of reversible change of oxidation state, nonbenzoid aromatic structure, low toxicity for mammal organisms and potential use as source of iron, make its derivatives ideal fragments for development of new drugs.

In the last two decades the ferrocenyl derivatives of purine and pyrimidine attracted high interest that might be explained by their unique features, in particular, redox properties, capability to penetrate cellar membranes, and low toxicity [3]. Different compounds were obtained, where fragments of ferrocene were in various positions of purine and pyrimidine systems [4].

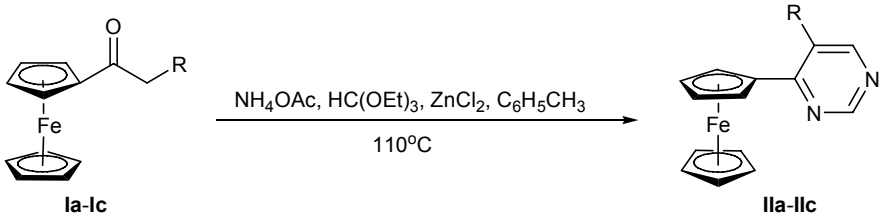
The ferrocenylpyrimidine derivatives possess high activity combined with low toxicity for various microorganisms, for example the ameba [5]. Such compounds may be used as initiators of radical reactions, allowing decreasing the polymerization temperature, increasing the reaction velocity, and obtaining polymers with higher thermal stability, for example, poly(methyl methacrylate) and polystyrene [6]. As a result the development of effective methods of synthesis of ferrocenyl-substituted pyrimidines became an actual task.

The first known way of 4-ferrocenylpyrimidine preparation is the reaction of ferrocenyllithium with pyrimidine [7]. Now several methods are known of substituted ferrocenylpyrimidine synthesis by the reactions of cross-coupling [8] or condensation of β-carbonyl compounds with urea or thiourea in the presence of Lewis acids [9] and also the condensation of the ferrocenyl ketones with the guanidine analogs [1, 10]. Among the defects of the described methods there are low yields, application of catalysts, the necessity of multistage synthesis in severe conditions [9].

We developed a convenient one-stage method of the preparation of 4,5-disubstitued ferrocenyl- and ferrocenoylpyrimidine **IIa–IIe** by $ZnCl_2$-cathalysed three-component condensation of ferrocenylcarbonyl compounds with triethyl orthoformate and ammonium acetate.

The initial acetylferrocene **Ia** and propionylferrocene **Ib** were obtained by acylation of ferrocene with the corresponding acid chlorides by Friedel-Krafts

Scheme 1.



Ia-Ic **IIa-IIc**

R = H (**a**), CH_3 (**b**), $COOCH_3$ (**c**).

reaction in dichloromethane using the aluminum chloride as the catalyst [11].

1,3-Dicarbonyl derivatives **Id** and **Ie** may be obtained by Claisen condensation of acetylferrocene **Ia** and corresponding esters in the presence of sodium ethylate [12, 13]. To increase the yield we used potassium *tert*-butoxide in benzene [14] that made it possible to simplify the isolation and purification of the intermediate potassium salt. After adding equimolar quantity of acetic acid to the salt suspension in methylene chloride 4,4,4-trifluoro-1-ferrocenyl-butane-1,3-dionee **Id** and ethyl ester of ferrocenyl-pyruvic acid **Ie** were obtained in an overall yield 70–89%. The presence in ^{1}H NMR spectra of compounds **Id** and **Ie** of broad singlets at 14.95 and 15.26 ppm (OH) and singlets (1H) at 6.54 (**Id**) and 6.09 ppm (**Ie**) proves that they exist in the solution in the enol form. Methyl ferrocenoylacetate (**Ic**) was obtained by the same method from acetylferrocene and dimethyl carbonate.

4,5-Disubstituted pyrimidines may be obtained by the [3+1+1+1]annulation and intermolecular cyclization [15]. 4,5-Disubstituted ferrocenylpyrimidines **IIa-IIc** were obtained in one stage by $ZnCl_2$-catalized three-component condensation of various functionalized ferrocenylcarbonyl compounds with triethyl orthoformate and ammonium acetate (Scheme 1).

If 4,4,4-trifluoro-1-ferrocenylbutane-1,3-dione is used as the initial compound then the reaction product would be [4-(trifluoromethyl)pyrimidin-5-yl](ferrocenyl)methanone **IId** (Scheme 2).

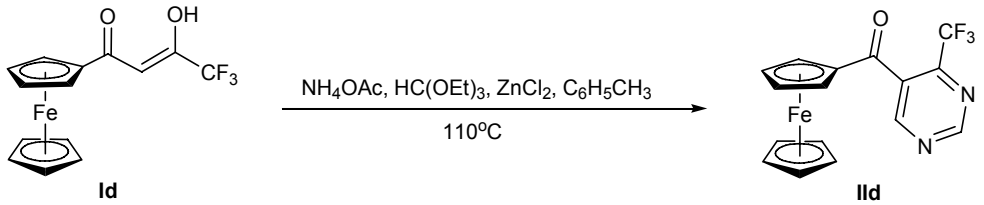
The structure of compound **IId** was established on the bases of on the data of ^{1}H and ^{13}C NMR and ^{1}H/^{13}C-heteronuclear correlations. In the ^{13}C NMR spectrum of the compound **IId** a singlet of carbon atom of the C=O group is present at 197 ppm, and not a quadruplet typical of 4-ferrocenyl-5-trifluoroacetylpyrimidine. Basing on this data we can state that the product of the reaction is [4-(trifluoromethyl) pyrimidin-5-yl](ferrocenyl)-methanone. If ethyl ferrocenoylpyruvate (**Ie**) is used in the same conditions the reaction proceeds in another way, and as a result the only isolated product of this process is ethyl 6-ferrocenyl-pyrimidine-4-carboxylate (**IIe**) (Scheme 3).

In the ^{1}H NMR spectra of the obtained compounds there is a number of signals belonging to the protons of the substituted and unsubstitued cyclopentadienyl rings (3.90–4.44 ppm), pyrimidine protons (6.54–7.13 ppm), and protons of the substituents. The assignment of the signals in the ^{13}C NMR spectra of the ferrocene derivatives was made basing on the HSQC and HMBC heteronuclear correlations.

The reaction of [3+1+1+1]annulation may be carried out with the subsequent intramolecular cyclization using various enamines [15] as initial compounds. In that manner the functionalized enamine **III** was obtained in a high yield, which was designed to be used for ethyl 5-ferrocenoylpyrimidine-4-carboxylate synthesis. Enamine **III** was obtained by interaction of ethyl ferrocenoylpyruvate (**Ie**) and methanol solution of ammonia in the presence of ammonium acetate (Scheme 4).

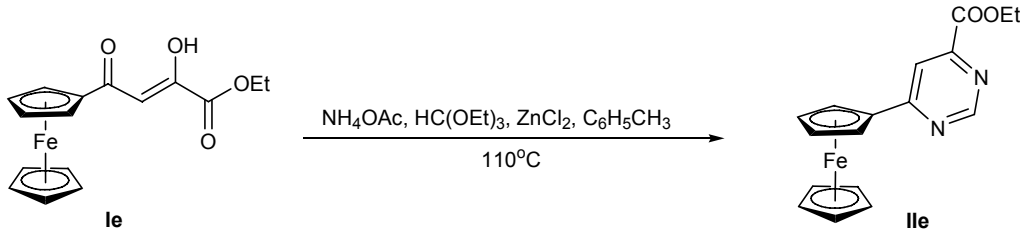
The pattern of signals in the ^{1}H NMR spectrum confirms that the reaction product is present in the enamine form. The broad singlets are present in the

Scheme 2.



Id **IId**

Scheme 3.



spectrum from the protons of the NH_2 group at 5 and 9 ppm, and a singlet in the area of 9 ppm evidences the formation of the hydrogen bond between NH and the carbonyl group.

Enamine **III** was introduced in the reaction of [3+1+1+1]annulation, however again the only isolated reaction product was ethyl 6-ferrocenyl-pyrimidine-4-carboxylate (**IIe**). At the attempt to synthesize compound **IIe** from the ethyl ferrocenoylpyruvate and the formamidine acetate only enamine **III** was obtained. Nowadays the obtained compounds are tested for the biological activity.

The electrochemical properties of pyrimidine derivatives of ferrocene were studied by the cyclic voltammetry. The study was carried out in acetonitrile in the presence of 0.05 M Bu_4NBF_4 on a platinum electrode using Ag/AgCl/KCl as a reference electrode.

The voltammograms of all studied compounds show the same reversible one electron redox-transfer, most likely corresponding to the oxidation of the ferrocene fragment. The oxidation potential of compound **IIc** is 680 mV. This potential is more anodic than the potential of unsubstitued ferrocene (E^{Ox} 500 mV) because of the influence of the electron-acceptor pyrimidine fragment. To compare the redox potentials by the known method *o*-tolylferrocene was obtained by reacting ferrocene with *o*-tolyldiazonium chloride [16]. Its redox potential (540 mV) shows that the pyrimidine ring possesses higher electon-acceptor properties than the benzene ring. The similar oxidation potentials of compounds **IIc** and **IIb** (675 mV) mean that the nature of the substituent in the pyrimidine fragment does not much affect the oxidation potential. The position of the ester group in the pyrimidine ring also does not change the value of the oxidation potential. The oxidation potential of compound **IIe** (685 mV) is more shifted in anoidic region comparing to the oxidation potential of compound **IIc** that can be caused by the influence of the acceptor carbonyl group located between the ferrocene and the pyrimidine fragment.

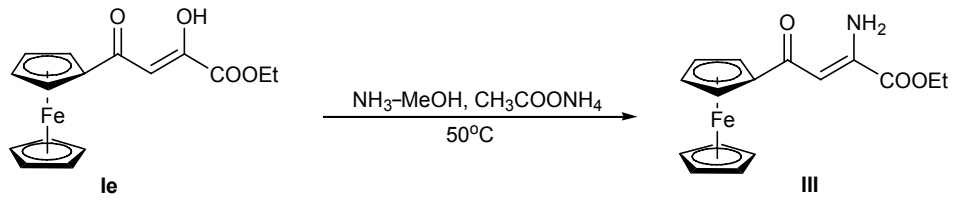
EXPERIMENTAL

The solvents were purified by standard methods and distillated in argon atmosphere just before use. The melting points of the substances were determined using Boëtius microblock. The EI mass spectra were recorded on the instrument Finnigan Polaris Q, temperature of the ionization chamber 250°C. The energy of the ionizing electrons 70 eV. ^1H and ^{13}C NMR spectra in deuterochloroform at 30°C were registered on Bruker DRX-500 instrument with working frequencies 500.13 and 125.76 MHz correspondingly. Chemical shifts are measured relatively to signals of residual protons of the solvent. The full correlation of the signals of all recorded NMR spectra was carried out by using NMR heteronuclear correlations (HSQC and HMBC) applying the gradient methods.

The Acros Organics reagents were used without preliminary purification.

1,3-Dicarbonyl compounds. General method [14]. To a suspension of 24.64 g (0.22 mol) of *t*-BuOK in 300 mL of benzene was added 45.6 g (0.2 mol) of acetylferrocene. To the mixture was added dropwise

Scheme 4.



0.22 mol of the corresponding ester. The reaction mixture was boiled while stirring for 2 h, then cooled to room temperature. The potassium salt was filtered off, washed with ether till colorless washings and dispersed in CH_2Cl_2. The suspension was treated with glacial acetic acid until pH 5 then the organic fraction was washed with water and brine, dried with anhydrous Na_2SO_4. The solvent was removed in a vacuum on a rotary evaporator.

Methyl ferrocenoylacetate (Ic). Yield 80%. Red crystals, mp 79–80°C. ^{1}H NMR spectrum, δ, ppm: 3.77 s (2H, CH_2), 3.79 s (3H, CH_3), 4.27 s (5H, Fc), 4.58 s (2H, Fc), 4.81 s (2H, Fc). ^{13}C NMR spectrum, δ, ppm: 46.7, 52.4, 69.7, 70.1, 73.0, 78.2, 168.0, 195.9. Mass spectrum, *m/z* ($I_{rel.}$,%): 286 $[M]^+$ (100).

4,4,4-Trifluoro-1-ferrocenylbutane-1,3-dione (Id). Yield 83%. Dark violet crystals, mp 100–101°C. ^{1}H NMR spectrum, δ, ppm: 4.24 s (5H, Fc), 4.69 s (2H, Fc), 4.87 s (2H, Fc), 6.09 s (1H, CH), 15.26 b.s (1H, OH). ^{13}C NMR spectrum, δ, ppm: 69.53, 71.25, 74.17, 75.66, 93.41, 120.34 q (CF_3, $^1J_{C,F}$ 279 Hz), 170.34 q (C^3, $^2J_{C,F}$ 35.5 Hz), 194.75. Mass spectrum, *m/z* ($I_{rel.}$, %): 324 $[M]^+$ (100).

Ethyl ferrocenoylpyruvate (Ie). Yield 89%. Dark violet crystals, mp 77°C (77°C [14]). ^{1}H NMR spectrum, δ, ppm: 1.38 t (3H, CH_3, *J* 7 Hz), 4.19 s (5H, Fc), 4.34 q (2H, CH_2, *J* 7 Hz), 4.62 s (2H, Fc), 4.85 s (2H, Fc), 6.54 s (1H, CH), 14.95 b.s (1H, OH). ^{13}C NMR spectrum, δ, ppm: 14.0, 62.2, 69.3, 70.5, 73.4, 77.4, 100.0, 162.6, 163.6, 197.7. Mass spectrum, *m/z* ($I_{rel.}$, %): 328 $[M]^+$ (62).

Ethyl 2-amino-3-ferrocenoylprop-3-enoate III. 5 mmol of ethyl ferrocenoylpyruvate was dissolved in 7 N ammonium methanol solution and 385 mg (5 mmol) of ammonium acetate was added. The reaction mixture was heated to 50°C at stirring. The course of the reaction was monitored by TLC. The solvent was removed in a vacuum on a rotary evaporator. Pure enamine **III** was obtained by chromatography of the residue on silica gel (eluent $CHCl_3$–MeOH, 9 : 1). Orange crystals. Yield 80%. ^{1}H NMR spectrum, δ, ppm: 1.40 t (3H, CH_3, *J* 6 Hz), 4.17 s (5H, Fc), 4.38 q (2H, CH_2, *J* 6 Hz), 4.47 s (2H, Fc), 4.82 s (2H, Fc), 5.72 b.s (1H, NH), 6.19 s (1H, CH), 9.06 b.s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 14.16, 62.50, 68.95, 69.95, 71.82, 81.69, 95.06, 144.77, 164.21, 195.83. Mass spectrum, *m/z* (I_{rel}, %): 327 $[M]^+$ (100). Found, %: C 57.94; H 5.32; Fe 16.84. $C_{16}H_{17}FeNO_3$. Calculated, %: C 58.74; H 5.24; Fe 17.07. *M* 327.06.

4- and 5-Ferrocenyl-substituted pyrimidine. General method. To a suspension of 6.8 mg (0.050 mmol) of $ZnCl_2$ and 220 mg (1.5 mmol) of triethyl orthoformate in toluene was added 0.5 mmol of the corresponding ketone and 77 mg (1.0 mmol) of ammonium acetate. The reaction mixture was heated to 110°C while stirring, the course of the reaction was monitored by TLC. The reaction mixture was treated with 5 mL of concentrated solution of $NaHCO_3$, the products of the reaction were extracted with $CHCl_3$, the combined organic extracts were dried with Na_2SO_4. The solvent was removed in a vacuum on a rotary evaporator. The residue was chromatographed on a column packed with silica gel (eluent ethyl acetate–hexane, 3 : 1).

4-Ferrocenylpyrimidine (IIa). Yield 58%. Orange oily substance. ^{1}H NMR spectrum, δ, ppm: 3.98 s (5H, Fc), 4.44 s (2H, Fc), 4.90 s (2H, Fc), 7.22 d (1H, CH, *J* 5.1 Hz), 8.44 d (1H, CH, *J* 5.4 Hz), 8.95 s (1H, CH). ^{13}C NMR spectrum, δ, ppm: 67.88, 70.00, 71.36, 79.67, 116.66, 155.90, 158.72, 167.94. Mass spectrum, *m/z* ($I_{rel.}$, %): 264 $[M]^+$ (100). Found, %: C 62.81; H 4.67; Fe 20.86. $C_{14}H_{12}FeN_2$. Calculated, %: C 63.67; H 4.58; Fe 21.15. *M* 264.04.

5-Methyl-4-ferrocenylpyrimidine (IIb). Yield 50%. Orange crystals, mp 70°C. ^{1}H NMR spectrum, δ, ppm: 2.47 s (1H, CH_3), 4.08 s (5H, Fc), 4.50 t (2H, Fc, *J* 2.1 Hz), 5.04 t (2H, Fc, *J* 2.1 Hz), 8.37 s (1H, CH), 8.91 s (1H, CH). ^{13}C NMR spectrum, δ, ppm: 18.28, 69.86, 70.21, 71.02, 77.16, 81.25, 126.76, 19.76156.29, 158.02, 165.77. Mass spectrum, *m/z* ($I_{rel.}$, %): 278 $[M]^+$ (100). Found, %: C 63.74; H 5.17; Fe. $C_{15}H_{14}FeN_2$. Calculated, %: C 64.78; H 5.07; Fe 20.08. *M* 278.05.

Methyl 5-ferrocenylpyrimidine-4-carboxylate (IIc). Yield 85%. Orange crystals, mp 151–152°C. ^{1}H NMR spectrum, δ, ppm: 3.93 s (3H, CH_3), 4.06 s (5H, Fc), 4.53 s (2H, Fc), 4.94 s (2H, Fc), 8.69 s (1H, CH), 9.07 s (1H, CH). ^{13}C NMR spectrum, δ, ppm: 29.7, 52.67, 70.02, 70.11, 70.34, 71.53, 156.43, 158.71, 166.08, 167.40. Mass spectrum, *m/z* ($I_{rel.}$, %): 322 $[M]^+$ (100). Found, %: C 58.86; H 4.51; Fe 17.02. $C_{16}H_{14}FeN_2O_2$. Calculated, %: C 59.65; H 4.38; Fe 17.34. *M* 322.04.

[4-(Trifluoromethyl)pyrimidin-5-yl](ferrocenyl) methanone (IId). Yield 70%. Orange oily substance. ^{1}H NMR spectrum, δ, ppm: 4.13 s (5H, Fc), 4.65 s (2H, Fc), 5.08 s (2H, Fc), 7.57 s (1H, CH), 9.16 s (1H, CH). ^{13}C NMR spectrum, δ, ppm: 68.41, 70.30, 72.24,

92.14, 121.80 q (C^5, $^3J_{C,F}$ 7.5 Hz), 129.80 q (CF_3, $^1J_{C,F}$ 279 Hz), 146.13 q (C^4, $^2J_{C,F}$ 32.5 Hz), 154.8, 159.00, 195.6. Mass spectrum, *m/z* ($I_{rel.}$,%): 360 $[M]^+$ (100). Found, %: C 52.71; H 3.18; Fe 15.32. $C_{16}H_{11}F_3FeN_2O$. Calculated, %: C 53.36; H 3.08; Fe 15.51. *M* 360.02.

Ethyl 6-ferrocenylpyrimidine-4-carboxylate (IIe). Yield 78%. Red oily substance. ^{1}H NMR spectrum, δ, ppm: 1.47–1.52 t (3H, CH_3, *J* 15 Hz), 4.09 s (5H, Fc), 4.52–4.55 q (2H, CH_2, *J* 9 Hz), 4.61 s (2H, Fc), 5.08 s (2H, Fc), 7.96 s (1H, CH), 9.17 s (1H, CH). ^{13}C NMR spectrum δ, ppm: 14.26, 62.63, 68.33, 70.21, 72.05, 79.06, 116.26, 153.78, 159.07, 164.63, 170.95, 195.72. Mass spectrum, *m/z* ($I_{rel.}$,%): 336 $[M]^+$ (100). Found, %: C 59.15; H 4.96; Fe 16.18. $C_{17}H_{16}FeN_2O_2$. Calculated, %: C 60.74; H 4.80; Fe 16.61. *M* 336.06.

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