Multi-component condensation of 4-hydroxy-6-methyl-1*H*-pyridin-2-one with carbonyl compounds and Meldrum´s acid*

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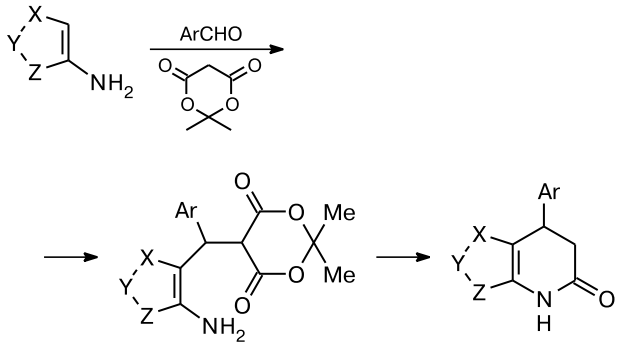
A convenient method for the synthesis of earlier unknown substituted derivatives of 4-aryl-7-methyl-4,6-dihydro-3*H*-pyrano[3,2-*c*]pyridine-2,5-dione and ethyl 3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)-3-arylpropionate was developed based on the multi-component condensation of 4-hydroxy-6-methyl-1*H*-pyridin-2-one, Meldrum´s acid, and carbonyl compounds.

Key words: multi-component condensation, Meldrum´s acid, 4-hydroxy-6-methyl-1*H*-pyridin-2-one.

Earlier, we have conducted systematic studies of the reaction of heterocyclic enamines **1** with carbonyl compounds and the Meldrum´s acid, which was the basis for development of a convenient general approach to the synthesis of fused heterocyclic systems **2** containing a dihydropyridinone fragment[1—5] (Scheme 1).

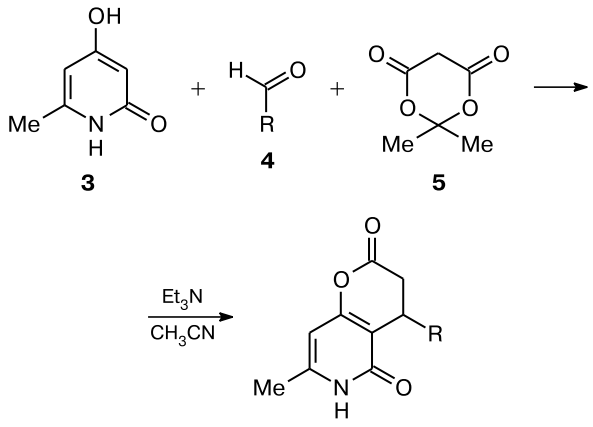
Scheme 1



We suggested that the use of enols, the oxygen analogs of enamines, as a nucleophilic component will make it possible to synthesize fused dihydropyranones possessing potential biological activity.[6,7] It should be noted that the reactivity of heterocyclic enols is considerably lower than that of enamines and not sufficient for the addition to the Meldrum´s acid arylmethylene derivatives. This makes it necessary to use a basic catalyst generating a enolate anion, which functions as a nucleophile in the Michael reaction.

The purpose of the present work is the studies of a multi-component condensation of 4-hydroxy-6-methyl-1*H*-pyridin-2-one **3** with carbonyl compounds and the Meldrum´s acid, as well as the studies of properties of the products formed. We have shown that the reaction of pyridinone **3** with aldehydes **4** and the Meldrum´s acid **5** in acetonitrile in the presence of triethylamine used as a base led to the good yields of 4-aryl-7-methyl-4,6-dihydro-3*H*-pyrano[3,2-*c*]pyridine-2,5-diones **6** (Scheme 2, Table 1).

Scheme 2



6: R = Ph (**a**), 2-MeO-C_6H_4 (**b**), 3-Br-C_6H_4 (**c**), 4-MeO-C_6H_4 (**d**), 3-MeO-4-HO-C_6H_3 (**e**), 3,4-$(MeO)_2$-C_6H_3 (**f**), 2,4,5-$(MeO)_3$-C_6H_2 (**g**), 2,3,4-$(MeO)_3$-C_6H_2 (**h**)

A plausible scheme of the transformation includes an initial condensation of aldehydes with the Meldrum´s acid,

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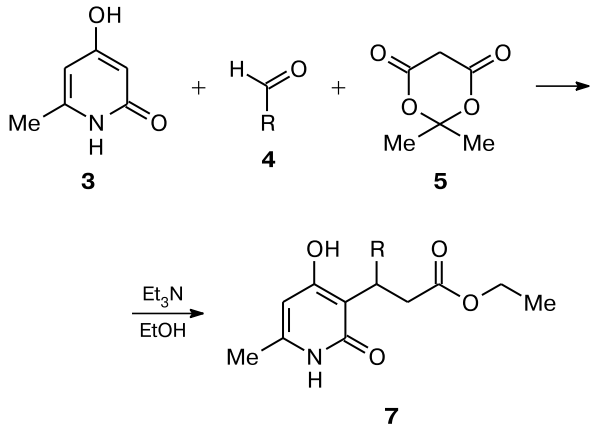
the formation and subsequent addition of the enolate anion by the Michael reaction, and a final intramolecular cyclization, which is accompanied by elimination of the CO_2 and acetone molecules. It should be noted that the solvent plays an important role in the reaction under study. Thus, the literature describes similar multi-component reactions involving aldehydes and the Meldrum´s acid carried out in alcohols.[8,9] We have shown that when the condensation is conducted in ethanol, the process did not stop on the step of the formation of products **6**, rather the pyranone ring opening takes place leading to esters **7** (Scheme 3, Table 2). Thus, two types of products can be synthesized in good yields depending on the solvent used: cyclic dihydropyranones **6** or open-chain esters **7**. Compounds **7** were also obtained from pyranones **6** upon treatment with triethylamine in ethanol. From the stated above, a conclusion can be made that dihydropyranones **6** are labile enough compounds, prone to the pyran ring opening in the reactions with nucleophiles.

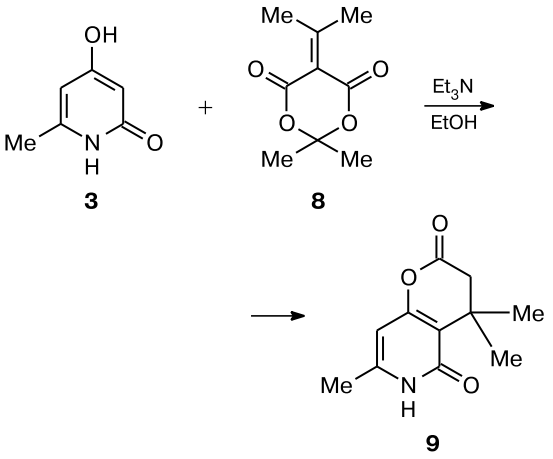
Scheme 3



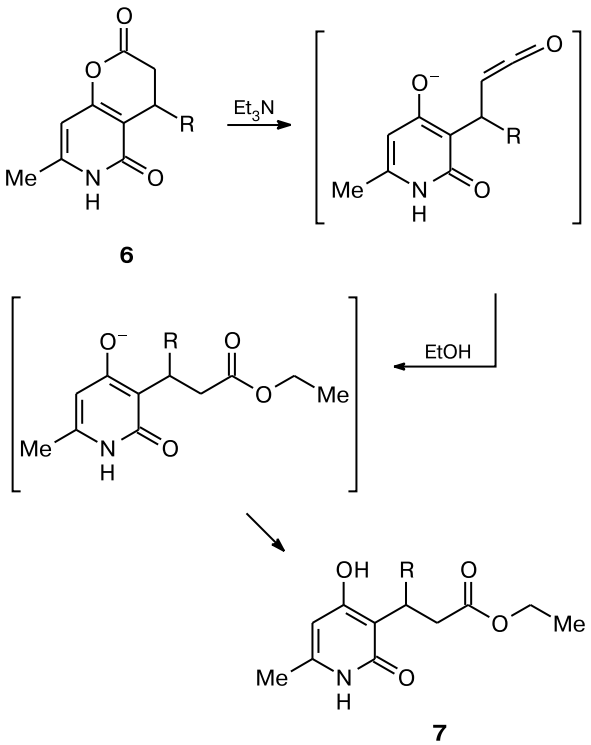
7: R = Ph (**a**), 2-MeO-C_6H_4 (**b**), 4-Cl-C_6H_4 (**c**), 4-MeO-C_6H_4 (**d**), 4-HO-C_6H_4 (**e**), 3-MeO-C_6H_4 (**f**), 2,3-$(MeO)_2$-C_6H_3 (**g**), 3,4,5-$(MeO)_3$-C_6H_2 (**h**)

The reactions considered above used various aldehydes as the carbonyl component. It is obvious that involvement of ketones in the process under study will make it possible to significantly broaden the scope of the suggested approach and vary the range of synthesized products. The literature described a three-component condensation of 2-aminopyrazole, acetone, and the Meldrum´s acid.[10] Initially, we attempted to use the same procedure with hydroxypyridone **3**, acetone, and the Meldrum´s acid and triethylamine as a base. However, this reaction resulted in the isolation of only the starting compound **3**. We suggested that the reason for the failure in the case of ketones was a low rate of the formation of the Meldrum´s acid ylidene derivative. Therefore, we preliminary synthesized 5-isopropylidene-2,2-dimethyl-[1,3]dioxane-4,6-dione (**8**), which was involved in the reaction with hydroxypyridone **3**. It should be noted that when an equimolar amount of compound (**8**) was used, the target product obtained contained a considerable amount of the starting compound **3** as an impurity. Apparently, this results from the decomposition of the ylidene derivative **8** in the process of the reaction. To solve this problem, we used a two-fold excess of compound **8**, that made it possible to obtain a pure target dihydropyranone **9** in good yield (Scheme 4).

Scheme 4



Scheme 5



It should be noted that independent from the type of the solvent used, only cyclic product **9** was formed in this case, that indicates the higher stability of the pyran ring in this compound as compared to the "aldehyde" derivative **6**. Based on these data, we can suggest two schemes for the transformation of dihydropyranones **6** to the open-chain esters **7**, which includes either direct nucleophilic attack by EtOH at the carbonyl carbon atom of the dihydropyran ring, or the generation of unstable ketene, which then adds an ethanol molecule with the formation of products **7** (Scheme 5).

In conclusion, we studied a multi-component condensation of 4-hydroxy-6-methyl-1*H*-pyridin-2-one with carbonyl compounds and the Meldrum´s acid and showed that depending on the solvent, two types of products can be synthesized: cyclic dihydropyranones **6** or open-chain esters **7**.

Experimental

^{1}H NMR spectra of were recorded on a Bruker AM 300 spectrometer (300 MHz) in DMSO-d_6. Melting points were measured on a Boetius heating stage and were not corrected.

4-Hydroxy-6-methyl-1*H*-pyridin-2-one (**3**) and 5-isopropylidene-2,2-dimethyl[1,3]dioxane-4,6-dione (**8**) were obtained using methods described in the literature.[11,12]

Synthesis of 4-aryl-7-methyl-4,6-dihydro-3*H*-pyrano[3,2-*c*]-pyridine-2,5-diones 6a—h (general procedure). A mixture of 4-hydroxy-6-methyl-1*H*-pyridin-2-one (0.25 g, 2 mmol), Meldrum´s acid (0.33 g, 2.3 mmol), the corresponding aldehyde (2.2 mmol), and triethylamine (0.23 g, 2.3 mmol) in acetonitrile (5 mL) was refluxed for 1 h and cooled, a precipitate formed was filtered off and washed with ethanol on the filter.

7-Methyl-4-phenyl-4,6-dihydro-3*H*-pyrano[3,2-*c*]pyridine-2,5-dione (6a). The yield was 66%, m.p. 273—275 °C. Found (%): C, 70.40; H, 5.20; N, 5.40. $C_{15}H_{13}NO_3$ (255.28). Calculated (%): C, 70.58; H, 5.13; N, 5.49.

4-(2-Methoxyphenyl)-7-methyl-4,6-dihydro-3*H*-pyrano-[3,2-*c*]pyridine-2,5-dione (6b). The yield was 81%, m.p. 279—281 °C. Found (%): C, 67.51; H, 5.18; N, 4.84. $C_{16}H_{15}NO_4$ (285.30). Calculated (%): C, 67.36; H, 5.30; N, 4.91.

4-(3-Bromophenyl)-7-methyl-4,6-dihydro-3*H*-pyrano[3,2-*c*]-pyridine-2,5-dione (6c). The yield was 76%, m.p. 292—294 °C. Found (%): C, 54.05; H, 3.48; N, 4.30. $C_{15}H_{12}BrNO_3$ (334.17). Calculated (%): C, 53.91; H, 3.62; N, 4.19.

4-(4-Methoxyphenyl)-7-methyl-4,6-dihydro-3*H*-pyrano-[3,2-*c*]pyridine-2,5-dione (6d). The yield was 59%, m.p. 251—253 °C. Found (%): C, 67.40; H, 5.19; N, 4.83. $C_{16}H_{15}NO_4$ (285.30). Calculated (%): C, 67.36; H, 5.30; N, 4.91.

4-(4-Hydroxy-3-methoxyphenyl)-7-methyl-4,6-dihydro-3*H*-pyrano[3,2-*c*]pyridine-2,5-dione (6e). The yield was 79%, m.p. 273—275 °C. Found (%): C, 63.57; H, 5.11; N, 4.60. $C_{16}H_{15}NO_5$ (301.30). Calculated (%): C, 63.78; H, 5.02; N, 4.65.

Table 1. ^{1}H NMR spectra (DMSO-d_6, d, *J*/Hz) of compounds **6a—h**

Compound	CH_3 (s, 3 H)	H—C—<u>H</u> (d, 1 H)	<u>H</u>—C—H (dd, 1 H)	H(4) (d, 1 H)	H(8) (s, 1 H)	R	NH (br.s, 1 H)
6a	2.23	2.83 (*J* = 15.9)	3.32 (*J* = 7.8; *J* = 15.9)	4.35 (*J* = 7.8)	6.35	7.1—7.45 (m, 5 H, C_6H_5)	11.72
6b	2.25	2.65 (*J* = 16.1)	3.25 (*J* = 8.3; *J* = 16.1)	4.45 (*J* = 8.3)	5.95	6.85 (m, 2 H, C_6H_4); 7.03 (m, 1 H, C_6H_4); 7.25 (m, 1 H, C_6H_4); 3.75 (s, 3 H, MeO)	11.65
6c	2.25	2.88 (*J* = 15.9)	3.34 (*J* = 8.1; *J* = 15.9)	4.35 (*J* = 8.1)	6.45	7.38 (s, 1 H, C_6H_4); 7.05 (d, 1 H, C_6H_4, *J* = 7.9); 7.30 (t, 1 H, C_6H_4, *J* = 7.9); 7.47 (d, 1 H, C_6H_4, *J* = 7.9)	11.95
6d	2.23	2.81 (*J* = 15.9)	3.32 (*J* = 6.8; *J* = 15.9)	4.3 (*J* = 6.8)	6.0	6.87 (d, 2 H, C_6H_4, *J* = 8.4); 7.05 (d, 2 H, C_6H_4, *J* = 8.4)	11.72
6e	2.20	2.80 (*J* = 16.0)	3.28 (*J* = 7.6; *J* = 16.0)	4.26 (*J* = 7.6)	6.0	6.82 (s, 1 H, C_6H_3); 6.68 (d, 1 H, C_6H_3, *J* = 8.0); 6.40 (d, 1 H, C_6H_3, *J* = 8.0); 3.71 (s, 3 H, MeO); 8.9 (br.s, 1 H, OH)	11.72
6f	2.22	2.89 (*J* = 15.8)	3.27 (*J* = 8.7; *J* = 15.8)	4.28 (*J* = 8.7)	6.0	6.90 (s, 1 H, C_6H_3); 6.50 (d, 1 H, C_6H_3, *J* = 8.2); 6.85 (d, 1 H, C_6H_3, *J* = 8.2); 3.7 (s, 3 H, MeO); 3.75 (s, 3 H, MeO)	11.75
6g	2.2	2.62 (*J* = 15.9)	3.23 (*J* = 7.7; *J* = 15.9)	4.32 (*J* = 7.7)	5.98	6.73 (s, 1 H, C_6H_2); 6.58 (s, 1 H, C_6H_2); 3.80 (s, 3 H, MeO); 3.73 (s, 3 H, MeO); 3.63 (s, 3 H, MeO)	—
6h	2.2	2.61 (*J* = 15.9)	3.31 (*J* = 8.4; *J* = 15.9)	4.4 (*J* = 8.4)	6.0	6.52 (d, 1 H, C_6H_2, *J* = 7.9); 7.88 (d, 1 H, C_6H_2, *J* = 7.9); 3.82 (s, 3 H, MeO); 3.76 (s, 3 H, MeO); 3.72 (s, 3 H, MeO)	11.63

4-(3,4-Dimethoxyphenyl)-7-methyl-4,6-dihydro-3*H*-pyrano-[3,2-*c*]pyridine-2,5-dione (6f). The yield was 75%, m.p. 250—252 °C. Found (%): C, 64.55; H, 5.33; N, 4.33. $C_{17}H_{17}NO_5$ (315.33). Calculated (%): C, 64.75; H, 5.43; N, 4.44.

4-(2,4,5-Trimethoxyphenyl)-7-methyl-4,6-dihydro-3*H*-pyrano-[3,2-*c*]pyridine-2,5-dione (6g). The yield was 64%, m.p. 256—258 °C. Found (%): C, 62.52; H, 5.62; N, 3.95. $C_{18}H_{19}NO_6$ (345.36). Calculated (%): C, 62.60; H, 5.55; N, 4.06.

4-(2,3,4-Trimethoxyphenyl)-7-methyl-4,6-dihydro-3*H*-pyrano-[3,2-*c*]pyridine-2,5-dione (6h). The yield was 78%, m.p. 267—269 °C. Found (%): C, 62.56; H, 5.64; N, 4.12. $C_{18}H_{19}NO_6$ (345.36). Calculated (%): C, 62.60; H, 5.55; N, 4.06.

Synthesis of ethyl 3-aryl-3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)propionates 7a—h (general procedure). A mixture of 4-hydroxy-6-methyl-1*H*-pyridin-2-one (0.25 g, 2 mmol), Meldrum´s acid (0.33 g, 2.3 mmol), the corresponding aldehyde (2.1 mmol), and triethylamine (0.23 g, 2.3 mmol) in ethanol (5 mL) was refluxed for 1 h and cooled, followed by addition of concentrated hydrochloric acid (0.35 g) and water until the reaction mixture grew opaque. A precipitate

Table 2. ^{1}H NMR spectra (DMSO-d_6, d, *J*/Hz) of compounds **7a—h**

Compound	$CH_3—CH_2$	CH_3 (s, 3 H)	H—C—H (dd, 1 H)	H—C—H (dd, 1 H)	CH (t, 1 H)	H(5) (s, 1 H)	R	OH (br.s, 1 H)	NH (br.s, 1 H)
7a	1.05 (t, 3 H, *J* = 7.1)	2.03	3.13 (*J* = 7.8; *J* = 15.5)	3.25 (*J* = 7.8; *J* = 15.5)	4.75 (*J* = 7.8)	5.62	7.0—7.40 (m, 5 H, C_6H_5)	10.20	10.90
7b	1.06 (t, 3 H, *J* = 7.1); 3.85 (q, 2 H, *J* = 7.1)	2.60	2.82 (*J* = 10.1; *J* = 15.1)	3.28 (*J* = 10.1; *J* = 15.1)	5.0 (*J* = 10.1)	5.65	6.7—6.9 (m, 2 H, C_6H_4); 7.05—7.25 (m, 2 H, C_6H_4); 3.78 (c, 3 H, MeO)	10.05	10.93
7c	1.05 (t, 3 H, *J* = 6.7); 3.98 (q, 2 H, *J* = 6.7)	2.10	3.10 (*J* = 7.3; *J* = 15.3)	3.30 (*J* = 7.3; *J* = 15.3)	4.73 (*J* = 7.3)	5.65	7.22 (d, 2 H, C_6H_4, *J* = 8.0); 7.38 (d, 2 H, C_6H_4, *J* = 8.0)	10.30	11.98
7d	1.05 (t, 3 H, *J* = 7.0); 3.95 (q, 2 H, *J* = 7.0)	2.05	3.08 (*J* = 8.1; *J* = 15.7)	3.20 (*J* = 8.1; *J* = 15.7)	4.68 (*J* = 8.1)	5.65	6.74 (d, 2 H, C_6H_4, *J* = 8.1); 7.27 (d, 2 H, C_6H_4, *J* = 8.1); 3.70 (s, 3 H, MeO)	10.18	10.90
7e	1.05 (t, 3 H, *J* = 7.1); 3.92 (q, 2 H, *J* = 7.1)	2.25	3.05 (*J* = 7.8; *J* = 15.4)	3.18 (*J* = 7.8; *J* = 15.4)	4.62 (*J* = 7.8)	5.65	7.12 (d, 2 H, C_6H_4, *J* = 8.3); 6.56 (d, 2 H, C_6H_4, *J* = 8.3); 9.33 (br.s, 1 H, OH)	10.14	10.90
7f	1.08 (t, 3 H, *J* = 7.1); 3.92 (q, 2 H, *J* = 7.1)	2.25	3.10 (*J* = 7.9; *J* = 15.6)	3.22 (*J* = 7.9; *J* = 15.6)	4.70 (*J* = 7.9)	5.65	6.62—7.15 (m, 4 H, C_6H_4); 3.68 (s, 3 H, MeO)	10.25	10.95
7g	1.20 (t, 3 H, *J* = 7.0); 3.95 (q, 2 H, *J* = 7.0)	2.05	2.81 (*J* = 7.0; *J* = 15.4)	3.25 (*J* = 7.0; *J* = 15.4)	5.25 (*J* = 7.0)	5.63	7.05 (m, 1 H, C_6H_3); 6.82 (m, 2 H, C_6H_3); 3.72 (s, 3 H, MeO) 3.78 (s, 3 H, MeO)	10.10	10.87
7h	1.08 (t, 3 H, *J* = 7.0); 3.92 (q, 2 H, *J* = 7.0)	2.05	3.15 (*J* = 7.9; *J* = 15.8)	3.22 (*J* = 7.9; *J* = 15.8)	4.68 (*J* = 7.9)	5.65	6.72 (s, 2 H, C_6H_2); 3.60 (s, 3 H, MeO); 3.70 (s, 6 H, 2MeO)	10.28	11.63

formed was filtered off and washed with aqueous ethanol on the filter.

Ethyl 3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)-3-phenylpropionate (7a). The yield was 66%, m.p. 184—186 °C. Found (%): C, 67.82; H, 6.24; N, 4.53. $C_{17}H_{19}NO_4$ (301.35). Calculated (%): C, 67.76; H, 6.36; N, 4.65.

Ethyl 3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)-3-(2-methoxyphenyl)propionate (7b). The yield was 82%, m.p. 214—216 °C. Found (%): C, 65.01; H, 6.49; N, 4.20. $C_{18}H_{21}NO_5$ (331.37). Calculated (%): C, 65.24; H, 6.39; N, 4.23.

Ethyl 3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)-3-(4-chlorophenyl)propionate (7c). The yield was 64%, m.p. 189—191 °C. Found (%): C, 60.62; H, 5.51; N, 10.65. $C_{17}H_{18}ClNO_4$ (335.79). Calculated (%): C, 60.81; H, 5.40; N, 10.56.

Ethyl 3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)-3-(4-methoxyphenyl)propionate (7d). The yield was 61%, m.p. 152—154 °C. Found (%): C, 63.13; H, 6.28; N, 4.22. $C_{18}H_{21}NO_5$ (331.37). Calculated (%): C, 65.24; H, 6.39; N, 4.23.

Ethyl 3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)-3-(4-hydroxyphenyl)propionate (7e). The yield was 57%, m.p. 220—222 °C. Found (%): C, 64.45; H, 5.92; N, 4.33. $C_{17}H_{19}NO_5$ (317.34). Calculated (%): C, 64.34; H, 6.03; N, 4.41.

Ethyl 3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)-3-(3-methoxyphenyl)propionate (7f). The yield was 67%, m.p. 189—191 °C. Found (%): C, 65.15; H, 6.41; N, 4.05. $C_{18}H_{21}NO_5$ (331.37). Calculated (%): C, 65.24; H, 6.39; N, 4.23.

Ethyl 3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)-3-(2,3-dimethoxyphenyl)propionate (7g). The yield was 54%, m.p. 182—184 °C. Found (%): C, 62.94; H, 6.52; N, 3.97. $C_{19}H_{23}NO_6$ (361.40). Calculated (%): C, 63.15; H, 6.41; N, 3.88.

Ethyl 3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)-3-(3,4,5-trimethoxyphenyl)propionate (7h). The yield was 40%, m.p. 133—135 °C. Found (%): C, 61.26; H, 6.33; N, 3.46. $C_{20}H_{25}NO_7$ (391.42). Calculated (%): C, 61.37; H, 6.44; N, 3.57.

Reaction of compounds 6 with ethanol (general procedure). A mixture of the corresponding 4-aryl-7-methyl-4,6-dihydro-3*H*-pyrano[3,2-*c*]pyridine-2,5-dione **6a**, **6b**, or **6d** (2 mmol) and triethylamine (0.4 g, 4 mmol) was refluxed in ethanol (7 mL) for 3 h and cooled, followed by addition of concentrated hydrochloric acid (0.5 g) and water until the reaction mixture grew opaque. A precipitate formed was filtered off and washed with aqueous ethanol on the filter. The yields of compounds **7a**, **7b**, and **7d** were 51, 63, and 45%, respectively. The ^{1}H NMR spectroscopy data showed that the compound obtained were identical to the products **7a**, **7b**, and **7d** synthesized according to the general procedure given above for the synthesis of ethyl 3-(4-hydroxy-6-methyl-2-oxo-1,2-dihydropyridin-3-yl)-3-arylpropionates **7a**—**h**.

Synthesis of 4,4,7-trimethyl-4,6-dihydro-3*H*-pyrano[3,2-*c*]-pyridine-2,5-dione (9). A mixture of 4-hydroxy-6-methyl-1*H*-pyridin-2-one (0.25 g, 2 mmol), 5-isopropylidene-2,2-dimethyl-[1,3]dioxane-4,6-dione (0.74 g, 4 mmol), and triethylamine (0.23 g, 2.3 mmol) in ethanol (5 mL) was refluxed for 1 h. Then, the reaction mixture was cooled, a precipitate formed was filtered off and washed with aqueous ethanol on the filter. The yield was 69%. M.p. 209—210 °C. ^{1}H NMR ([$^2H_6$]DMSO), δ: 1.31 (s, 6 H, 2 CH_3); 2.12 (s, 3 H, CH_3); 2.66 (s, 2 H, CH_2); 5.82 (s, 1 H, H(8)); 11.46 (br.s, 1 H, NH). Found (%): C, 63.57; H, 6.21; N, 6.65. $C_{11}H_{13}NO_3$ (207.23). Calculated (%): C, 63.76; H, 6.32; N, 6.76.

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