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SYNTHESIS OF SOME FUNCTIONALIZED PYRIDONES DERIVATIVES

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It was established formation of new substituted pyridone derivatives in terms of addition reaction according to Michael with the participation of 2-cyano-3-(4-pyridyl) acrylamide and benzoylacetone or ethyl alcohol in ether of acetic acid in the presence (MP) in methanol medium. The boiling of ethyl 5-cyano-2-hydroxy-2-methyl-6-oxo-4-phenylpiperidine-3-carboxylate in ethanol during 4 hours followed by dehydration and dehydrogenation (by air oxidation) generated 5-cyano-2-methyl-6-oxo-4-phenyl-1,6-dihydropyridine-3-carboxylic acid. Under similar conditions there were synthesized appropriate products of substituted pyridone according to Michael' addition reaction through interacting of 4-bromobenzoylacetone nitrile with 2-cyano-3-(2-fluorophenyl)acrylamide. Structures of all synthesized compounds were proven by NMR spectroscopy.

Keywords: 2-cyano-3-(4-pyridyl)acrylamide, fluorobenzylidenecyanoacetamide, 4-chloroacetoacetate, 4-bromobenzoylacetone nitrile

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Introduction

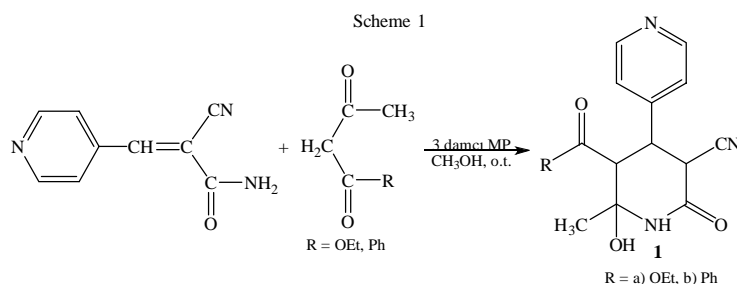
2-Pyridone, tautomer of 2-hydroxypyridine, is one of the major heteroaromatic rings in natural products, bioactive molecules and pharmaceutical ingredients. Ciclopirox, milrinone, camptothecin, (-)-citisin, fredericamycin, perampanel, bioactive molecules and pharmaceutical ingre-

dients contain valuable pyridone fragments in their composition. Also, synthesis methods of these compounds are different from in reaction conditions in literature [1-12]. Some articles [13-17] research into antimicrobial activity of pyridone derivatives.

Results and discussion

In the presence of methylpiperazine and methanol medium according to Michael addition reaction of benzoylacetone or ethyl

acetoacetate, 2-cyano-3-(pyridine-4-yl) acrylamide at a room temperature was hydroxysubstituted pyridone derivatives.

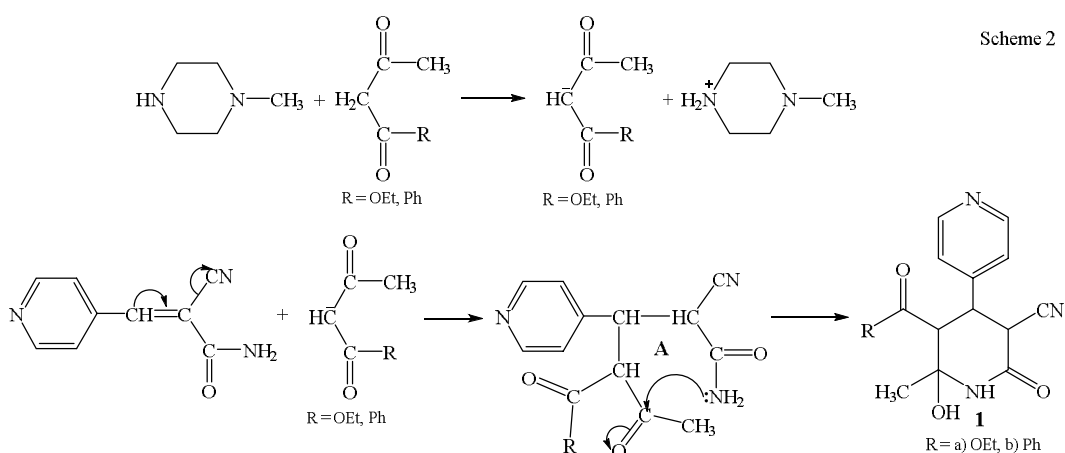


Scheme 1. Reaction of 2-cyano-3-(4-pyridyl)acrylamide with benzoylacetone and ethyl acetoacetate.

In our view, in the initial step of reaction the nucleophilic attack methylpiperazine to methylene-active compound results in the formation of corresponding anion

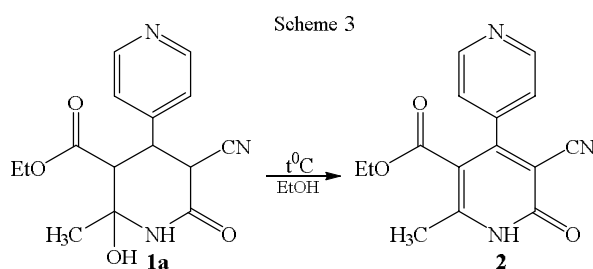
(nucleophilic particle), which, in turn, drew to the CH-electrophilic center of the activated double bond to form intermediate A (according to Michael adduct). In the final

step, in the intermediate **A**, the amide nitrogen desired ring closure product – pyridione attacked the carbonyl group to generate a (Scheme 2).

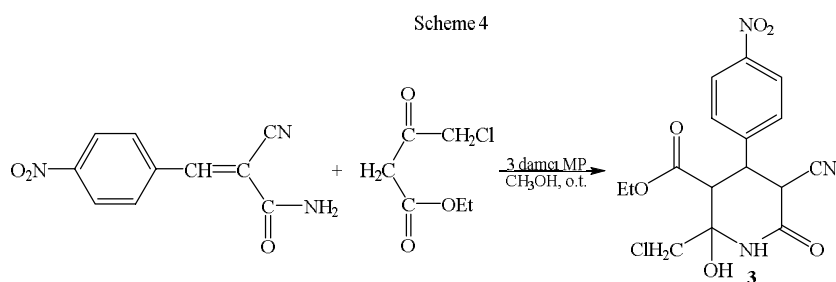


Boiling of ethyl 5-cyano-2-hydroxy-2-methyl-6-oxo-4-phenylpiperidine-3-carboxylate in ethanol for 4 hours was followed by dehydration and dehydrogenation (by air

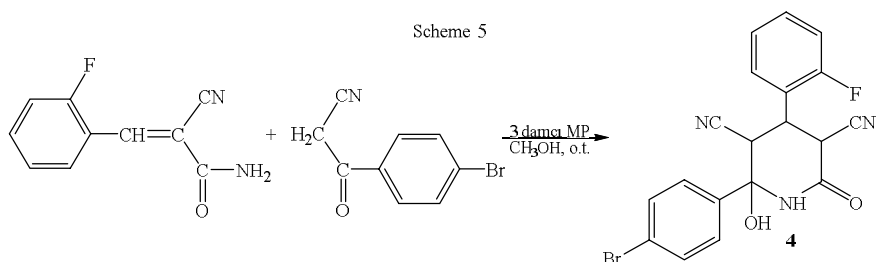
oxidation) to provide 5-cyano-2-methyl-6-oxo-4-phenyl-1,6-dihydropyridine-3-carboxylic acid.



Scheme 2. Synthesis of ethyl 5-cyano-2-methyl-6-oxo-4-phenyl-1,6-dihydropyridine-3-carboxylate



Scheme 3. Reaction of 2-cyano-3-(4-nitrophenyl)acrylamide with ethyl 4-chloroacetoacetate.



Scheme 4. Reaction of 2-cyano-3-(2-fluorophenyl)acrylamide with 4-bromobenzoylacetonitrile.

Under the same reaction condition, the Michael addition of 4-bromobenzoyl-acetonitrile to 2-cyano-3-(2-fluorophenyl)

acrylamide led to the formation of appropriate hydroxy substituted pyridone derivatives.

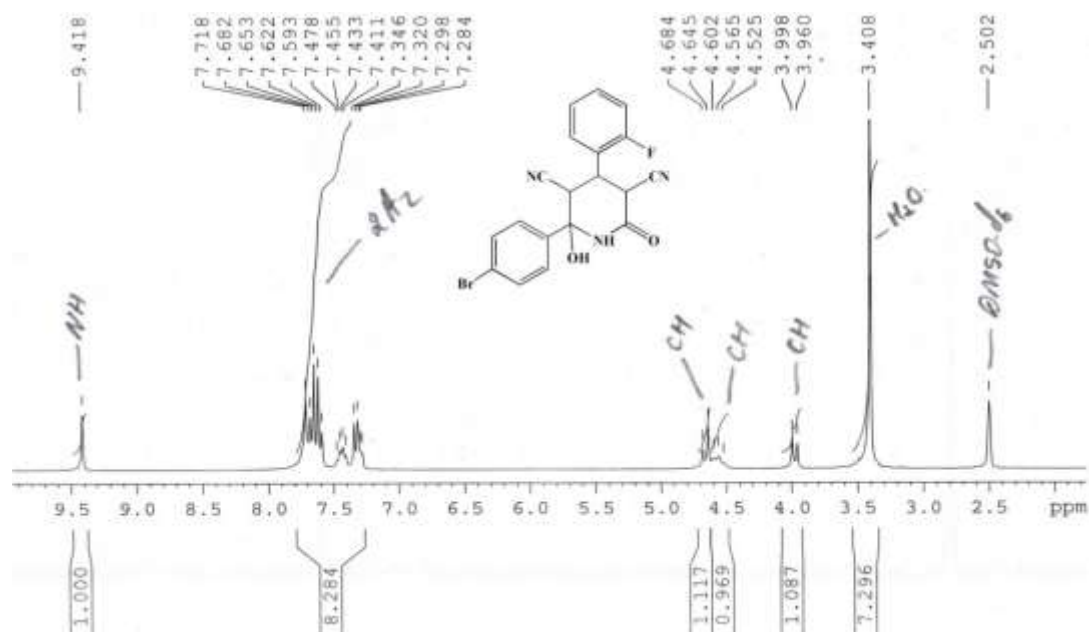


Fig. 1. ^1H NMR spectrum of 2-(4-bromophenyl)-4-(2-fluorophenyl)-2-hydroxy-6-oxopiperidine-3,5-dicarbonitrile (**4**)

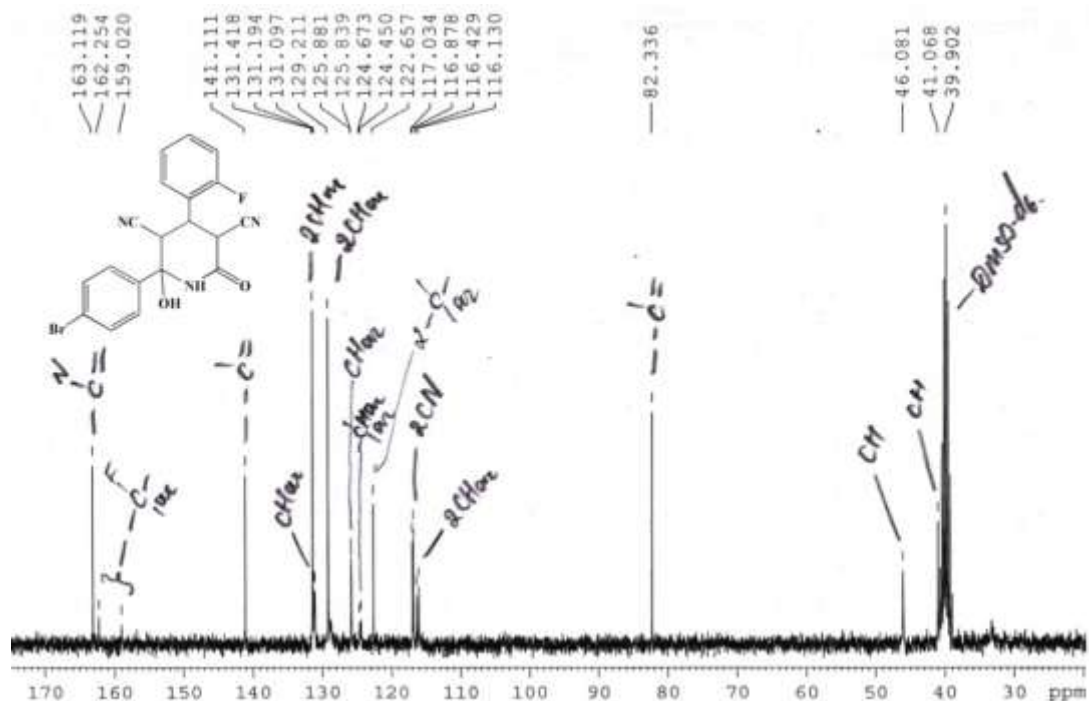


Fig. 2. ^{13}C NMR spectrum of 2-(4-bromophenyl)-4-(2-fluorophenyl)-2-hydroxy-6-oxopiperidine-3,5-dicarbonitrile (**4**)

Experimental part. General remarks

All commercially available chemicals were obtained from Merck and Fluka (Sigma - Aldrich) companies and used without further purification. Melting points were measured by Stuart SMP30 apparatus without correction. ^1H , ^{13}C NMR spectra (Fig.1) were recorded on BrukerAvance 300-MHz spectrometer at 300 and 75 MHz, respectively. Thin-layer chromatography (TLC) on commercial aluminum-backed plates of silica gel (60 F254) was used to monitor the reaction course.

Experimental procedures:

Ethyl 5-cyano-2-hydroxy-2-methyl-6-oxo-4-(pyridin-4-yl)piperidine-3-carboxylate (1a): 2-Cyano-3-(4-pyridyl)acrylamide (5.1 mmol) and ethyl acetoacetate (5.2 mmol) was stirred in 35 ml of methyl alcohol. Then 3-4 drops of 1-methylpiperazine were added to reaction mixture and stirred for 5 minutes. Then reaction mixture was held out at a room temperature for 48 h. Reaction course was monitored by TLC (EtOAc/n-hexane, 3:1). Crystals were precipitated after evaporation of solvent, filtered, recrystallized from ethanol-water mixture and obtained in pure form (yield 1.27 g, 82.47%). $T_{\text{mp.}} = 173^\circ\text{C}$.

^1H NMR (300 MHz, DMSO- d_6): 0.84 (t, 3H, CH_3 , $^3J_{\text{H-H}} = 7$); 1.47 (s, 3H, CH_3); 3.44 (d, 1H, CH, $^3J_{\text{H-H}} = 12.4$); 3.82 (k, 2H, CH_2O , $^3J_{\text{H-H}} = 6.9$); 3.92 (t, 1H, CH-Ar , $^3J_{\text{H-H}} = 12.3$); 4.31 (d, 1H, CH, $^3J_{\text{H-H}} = 12.6$); 6.27 (s, 1H, OH); 7.39 (d, 2H, $2\text{CH}_{\text{pyrid.}}$, $^3J_{\text{H-H}} = 5.1$); 8.55 (d, 2H, $2\text{CH}_{\text{pyrid.}}$, $^3J_{\text{H-H}} = 5.1$); 8.97 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO- d_6): 14.02 (CH_3CH_2), 28.34 (CH_3), 40.41 (CH-CN), 41.44 (CH-Ar), 54.24 (CH-CO_2), 60.64 (CH_2O), 80.72 ($\text{O-C}_{\text{dördli}}$), 117.29 (CN), 123.85 ($2\text{CH}_{\text{pyrid.}}$), 148.63 ($\text{C}_{\text{pyrid.}}$), 150.33 ($2\text{CH}_{\text{pyrid.}}$), 162.56 (N-C=O), 168.58 (O-C=O).

Found, %: 59.35 C; 5.67 H; 13.81 N. $\text{C}_{15}\text{H}_{17}\text{N}_3\text{O}_4$. Calculated, %: 59.40 C; 5.61 H; 13.86 N.

5-Benzoyl-6-hydroxy-6-methyl-2-oxo-4-(pyridin-4-yl)piperidine-3-carbonitrile (1b): 2-Cyano-3-(4-pyridyl)acrylamide (5.1 mmol) and benzoylacetone (5.2 mmol) was stirred in 35 ml of methyl alcohol. After 3-4 drops of 1-methylpiperazine added to reaction mixture and stirred for 5 minutes. Then reaction

mixture was held out at a room temperature for 48 h. Reaction course was monitored by TLC (EtOAc/n-hexane, 3:1). Crystals were precipitated after evaporation of solvent, filtered, recrystallized from ethanol-water mixture and obtained in pure form (yield 1.32 g, 77.65%). $T_{\text{mp.}} = 192^\circ\text{C}$.

^1H NMR (300 MHz, DMSO- d_6): 1.35 (s, 3H, CH_3); 3.86 (t, 1H, CH-Ar , $^3J_{\text{H-H}} = 11.4$); 4.60 (d, 1H, CH, $^3J_{\text{H-H}} = 12.3$); 4.68 (d, 1H, CH, $^3J_{\text{H-H}} = 11.1$); 6.46 (s, 1H, OH); 7.44-8.44 (m, 9H, $5\text{Ar-H}+4\text{CH}_{\text{pyrid.}}$); 8.84 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO- d_6): 27.01 (CH_3), 40.78 (CH-CN), 42.98 (CH-Ar), 55.15 (CH-C=O), 82.86 ($\text{O-C}_{\text{dördli}}$), 117.26 (CN), 123.83 ($2\text{CH}_{\text{arom.}}$), 129.04 ($2\text{CH}_{\text{pirid.}}$), 129.14 ($2\text{CH}_{\text{arom.}}$), 134.09 ($\text{CH}_{\text{arom.}}$), 138.17 ($\text{C}_{\text{ar.}}$), 148.52 ($\text{C}_{\text{pyrid.}}$), 150.25 ($2\text{CH}_{\text{pyrid.}}$), 162.43 (N-C=O), 199.23 (C=O).

Found, %: 68.00 C; 5.02 H; 12.60 N. $\text{C}_{19}\text{H}_{17}\text{N}_3\text{O}_3$. Calculated, %: 68.06 C; 5.07 H; 12.54 N.

Ethyl 5-cyano-2-methyl-6-oxo-1,6-dihydro-[4,4'-bipyridine]-3-carboxylate (2): Ethyl 5-cyano-2-hydroxy-2-methyl-6-oxo-4-(pyridin-4-yl)piperidine-3-carboxylate (5.1 mmol) was dissolved in 35 ml of ethyl alcohol and 5 h refluxed. Then the resulting reaction mixture was placed in a glass. Crystals were precipitated after evaporation of solvent, filtered, recrystallized from ethanol-water mixture and obtained in pure form (yield 1.07 g, 74.30%). $T_{\text{mp.}} = 147^\circ\text{C}$.

^1H NMR (300 MHz, DMSO- d_6): 0.70 (t, 3H, CH_3 , $^3J_{\text{H-H}} = 7.2$); 2.45 (s, 3H, CH_3); 3.83 (k, 2H, CH_2O , $^3J_{\text{H-H}} = 7$); 7.36 (d, 2H, $2\text{CH}_{\text{pyrid.}}$, $^3J_{\text{H-H}} = 5.1$); 8.71 (d, 2H, $2\text{CH}_{\text{pyrid.}}$, $^3J_{\text{H-H}} = 5.1$); 12.82 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO- d_6): 13.37 (CH_3CH_2), 19.13 ($\text{CH}_3\text{-C=}$), 61.43 (CH_2O), 101.07 ($=\text{C}_{\text{dördli}}$), 111.09 ($=\text{C}_{\text{dördli}}$), 115.42 (CN), 122.35 ($2\text{CH}_{\text{pyrid.}}$), 144.51 ($\text{C}_{\text{pyrid.}}$), 150.25 ($2\text{CH}_{\text{pyrid.}}$), 155.45 ($=\text{C}_{\text{dördli}}$), 157.53 ($=\text{C}_{\text{dördli}}$), 160.00 (N-C=O), 164.64 (O-C=O).

Found, %: 63.55 C; 4.65 H; 14.89 N. $\text{C}_{15}\text{H}_{13}\text{N}_3\text{O}_3$. Calculated, %: 63.60 C; 4.59 H; 14.84 N.

Ethyl 2-(chloromethyl)-5-cyano-2-hydroxy-4-(4-nitrophenyl)-6-oxopiperidine-3-carbo-

xylate (3): 2-Cyano-3-(4-nitrophenyl)acrylamide (5.1 mmol) and ethyl 4-chloroacetoacetate (5.2 mmol) stirred in 35 ml of methyl alcohol. After 3-4 drops of 1-methylpiperazine added to reaction mixture and stirred for 5 minutes. Then reaction mixture hold out at room temperature for 48 h. Reaction course was monitored by TLC (EtOAc/n-hexane, 3:1). Crystals were precipitated after evaporation of solvent, filtered, recrystallized from ethanol-water mixture and obtained in pure form (yield 1.57 g, 80.51%). $T_{mp.} = 209^{\circ}\text{C}$.

$^1\text{H NMR}$ (300 MHz, $\text{DMSO-}d_6$): 0.86 (t, 3H, CH_3 , $^3J_{\text{H-H}} = 6.9$); 3.68 (d, 1H, CH , $^3J_{\text{H-H}} = 12.3$); 3.69 (s, 3H, CH_2Cl); 3.85 (k, 2H, CH_2O , $^3J_{\text{H-H}} = 7$); 4.10 (t, 1H, CH-Ar , $^3J_{\text{H-H}} = 12.3$); 4.63 (d, 1H, CH , $^3J_{\text{H-H}} = 12$); 7.12 (s, 1H, OH); 7.70 (d, 2H, $2\text{CH}_{\text{arom.}}$, $^3J_{\text{H-H}} = 8.4$); 8.24 (d, 2H, $2\text{CH}_{\text{arom.}}$, $^3J_{\text{H-H}} = 8.4$); 9.10 (s, 1H, NH). $^{13}\text{C NMR}$ (75 MHz, $\text{DMSO-}d_6$): 13.97 (CH_2CH_2), 40.13 (CH-CN), 41.31 (CH-Ar), 48.16 (CH_2Cl), 49.80 (CH-COO), 61.06 (CH_2O), 83.03 ($\text{O-C}_{\text{dördli}}$), 117.01 (CN), 124.13 ($3\text{CH}_{\text{arom.}}$), 130.21 ($\text{CH}_{\text{arom.}}$), 147.10 ($\text{C}_{\text{ar.}}$), 147.47 ($\text{C}_{\text{ar.}}$), 163.30 (N-C=O), 167.71 (O-C=O).

Found, %: 50.39 C; 4.14 H; 11.07 N. $\text{C}_{16}\text{H}_{16}\text{N}_3\text{O}_6\text{Cl}$. Calculated, %: 50.33 C; 4.19 H; 11.02 N.

2-(4-Bromophenyl)-4-(2-fluorophenyl)-2-hydroxy-6-oxopiperidine-3,5-dicarbonitrile (4): 2-Cyano-3-(2-fluorophenyl)prop-2-enamide (5.1 mmol) and 4-bromobenzoylacetone nitrile (5.2 mmol) stirred in 35 ml of methyl alcohol. After 3-4 drops of 1-methylpiperazine were added to reaction mixture and stirred for 5 minutes. Then reaction mixture were held out at a room temperature for 48 h. Reaction course was monitored by TLC (EtOAc/n-hexane, 3:1). Crystals were precipitated after evaporation of solvent, filtered, recrystallized from ethanol-water mixture and obtained in pure form (yield 1.77 g, 84.28%). $T_{mp.} = 121^{\circ}\text{C}$.

$^1\text{H NMR}$ (300 MHz, $\text{DMSO-}d_6$): 3.98 (d, 1H, CH , $^3J_{\text{H-H}} = 11.4$); 4.56 (t, 1H, CH , $^3J_{\text{H-H}} = 11.5$); 4.66 (d, 1H, CH , $^3J_{\text{H-H}} = 11.7$); 7.28-7.72 (m, 8H, 8Ar-H+OH); 9.42 (s, 1H, NH). $^{13}\text{C NMR}$ (75 MHz, $\text{DMSO-}d_6$): 39.90 (CH-CN), 41.07 (CH-Ar), 46.08 (CH-CN), 82.34 ($\text{O-C}_{\text{dördli}}$), 116.13 ($\text{CH}_{\text{arom.}}$), 116.43 ($\text{CH}_{\text{arom.}}$), 116.88 (CN), 117.03 (CN), 122.66 ($\text{Br-C}_{\text{ar.}}$), 124.45-124.67 ($\text{C}_{\text{ar.}}$), 125.88 ($\text{CH}_{\text{arom.}}$), 129.21 ($2\text{CH}_{\text{arom.}}$), 131.10-131.19 ($\text{CH}_{\text{arom.}}$), 131.42 ($2\text{CH}_{\text{arom.}}$), 141.11 ($\text{C}_{\text{ar.}}$), 159.02-162.25 ($\text{F-C}_{\text{ar.}}$), 163.12 (N-C=O).

Found, %: 55.01 C; 3.20 H; 10.09 N. $\text{C}_{19}\text{H}_{13}\text{N}_3\text{FBrO}_2$. Calculated, %: 55.07 C; 3.14 H; 10.14 N.

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BƏZİ FUNKSIONALLAŞMIŞ PİRİDON TÖRƏMƏLƏRİNİN SİNTEZİ

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Metanol mühitində, metilpiperazinin (MP) iştirakında və otaq temperaturunda 2-siano-3-(4-piridil)akrilamid ilə benzoilasetonun və ya asetosirkə turşusunun etil efirinin Mixael birləşmə reaksiyasından müvafiq yeni əvəzlənmiş piridon törəmələrinin əmələ gəldiyi müəyyən edilmişdir. 5-Siano-2-hidroksi-2-metil-6-okso-4-fenilpiperidin-3-karboksil turşusunun etil efirinin 4 saat etil

spirtində qaynadılmasından 5-siano-2-metil-6-okso-4-fenil-1,6-dihidropiridin-3-karboksil turşusunun etil efiri əmələ gəlmişdir. Eyni reaksiya şəraitində 4-brombenzoilasetonitrilin 2-siano-3-(2-flüorfenil)akrilamidə Mixael birləşmə reaksiyasından müvafiq piridon törəməsi alınmışdır. Həmçinin, eyni reaksiya şəraitində 2-siano-3-(4-nitrofenil)akrilamid ilə 4-xlorasetoasetat turşusunun etil efirinin qarşılıqlı təsir reaksiyasından uyğun piridon törəməsi sintez edilmişdir. Sintez edilmiş bütün birləşmələrin quruluşları NMR spektroskopiyasının köməyi ilə təsdiq edilmişdir.

Açar sözlər: 2-siano-3-(4-piridil)akrilamid, flüorbenzilizidensianoasetamid, 4-xlorasetoasetat efiri, 4-brombenzoilasetonitril

СИНТЕЗ ПРОИЗВОДНЫХ НЕКОТОРЫХ ФУНКЦИОНАЛИЗИРОВАННЫХ ПИРИДОНОВ

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Выявлено образование новых производных замещенных пиридонов в условиях реакции присоединения по Михаэлю при участии 2-циано-3-(4-пиридил)акриламида и бензоилацетона или этилового эфира ацетоуксусной кислоты в присутствии метилпиперазина (MP) в среде метанола. При кипячении в этиловом спирте этилового эфира 5-циано-2-гидрокси-2-метил-6-оксо-4-фенилтиперидин-3-карбоновой кислоты в течение 4 часов образуется этиловый эфир 5-циано-2-метил-6-оксо-4-фенил-1,6-дигидропирид-3-карбоновой кислоты. В аналогичных условиях были синтезированы соответствующие продукты замещенных пиридонов по реакции присоединения Михаэля взаимодействием 4-бромбензоилацетонитрила с 2-циано-3-(2-фторфенил)акриламидом и 2-циано-3-(4-нитрофенил)акриламида с этилового эфира 4-хлорацетоуксусной кислоты. Структуры всех синтезированных соединений доказаны методом ЯМР-спектроскопии.

Ключевые слова: 2-циано-3-(4-пиридил)акриламид, фторбензилиденцианоацетамид, 4-хлорацетоуксусный эфир, 4-бромбензоилацетонитрил