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SYNTHESIS OF 3-SUBSTITUTED-PYRIDO[2,3-*d*]PYRIMIDINONES BY THE SUZUKI-MIYAURA METHOD

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Abstract. Initially, polymethylene [2,3-*d*]pyrimidinone derivatives were synthesized using known cyclization methods. The 3-bromo-tri, -tetra and –pentamethylene [2,3-*d*]pyrimidinones obtained in this step were subjected to Suzuki-Miyaura-type coupling reactions with aromatic boronic acids in the presence of a palladium catalyst in alkaline medium. As a result of this process, six new 3-substituted pyrimidinone derivatives were synthesized. The structures of the obtained compounds were reliably confirmed on the basis of ^{1}H NMR, ^{13}C NMR, mass spectral analysis and melting point (MP) data.

Keywords: aromatic boronic acids, 1,4-dioxane, dichloromethane, sodium carbonate, tetrakis(triphenylphosphine)palladium.

Suzuki-miyaura usulida 3-almashingan-pirido[2,3-*d*]pirimidinonlarning sintezi

Annotatsiya. Dastlab, polimetilen [2,3-*d*]pirimidinon hosilalari ma’lum siklizatsiya usullari yordamida sintez qilindi. Ushbu bosqichda olingan 3-bromo-tri, -tetra va –pentametilen [2,3-*d*]pirimidinonlar ishqoriy muhitda palladiy katalizatori ishtirokida aromatik boronik kislotalari bilan Suzuki-Miyaura tipidagi o‘zaro bog‘lanish reaksiyalariga kiritildi. Ushbu jarayon natijasida oltita yangi 3-almashgan pirimidinon hosilalari sintez qilindi. Olingan birikmalarning tuzilishi ^{1}H NMR, ^{13}C NMR, massa spektral tahlili va erish nuqtasi (MP) ma’lumotlari asosida ishonchli tarzda tasdiqlandi.

Kalit so‘zlar: aromatik boronik kislotalari, 1,4-dioksan, dixlormetan, natriy karbonat, tetrakis(trifenilfosfin)palladiy.

Синтез 3-замещенных-пиридо[2,3-*d*]пиримидинонов методом Сузуки-Мияуры

Аннотация. Первоначально производные полиметилен[2,3-*d*]пиримидинона были синтезированы с использованием известных методов циклизации. Полученные на этом этапе 3-бром-три-, -тетра- и -пентаметилен[2,3-*d*]пиримидиноны подвергали реакциям сочетания типа Сузуки-Мияуры с ароматическими борными кислотами в присутствии палладиевого катализатора в щелочной среде. В результате этого процесса были синтезированы шесть новых 3-замещенных производных пиримидинона. Структуры полученных соединений были достоверно подтверждены на основе данных ЯМР ^{1}H, ЯМР ^{13}C, масс-спектрального анализа и данных о температуре плавления (ТП).

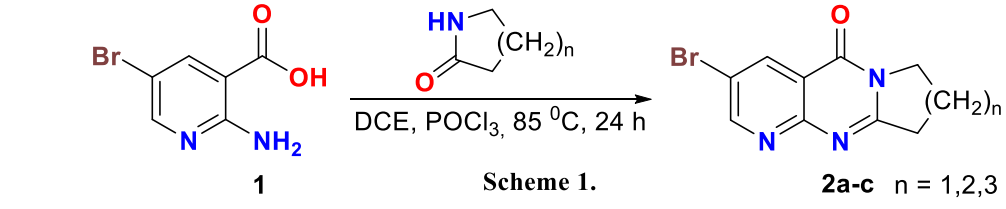
Ключевые слова: ароматические бороновые кислоты, 1,4-диоксан, дихлорметан, карбонат натрия,

тетраксис(трифенилфосфин)палладий.

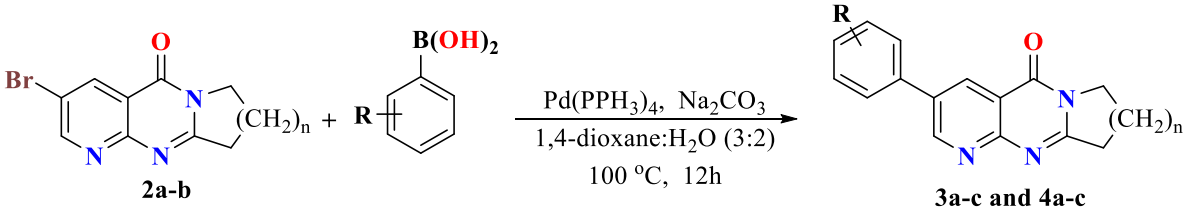
Introduction. Contemporary pharmaceutical research is intensively focused on the discovery and development of novel therapeutic agents targeting a wide spectrum of pathological conditions [1]. However, despite significant progress, the insufficient availability of effective pharmacological interventions—particularly for neurodegenerative disorders—remains a major global health challenge, resulting in millions of deaths annually [2] [3]. Clinically, the disease manifests through pronounced memory loss, deterioration of cognitive performance, and impairment of higher-order thinking processes [4][5]. Current epidemiological data indicate that tens of millions of individuals worldwide are affected by Alzheimer's disease, and prognostic models suggest a dramatic increase in patient numbers over the coming decades [6] [7] [8]. Researchers have proposed several theories to explain the development of Alzheimer's disease. To elucidate the underlying pathogenesis of this disorder, researchers have proposed multiple mechanistic hypotheses. Notably, the cholinergic deficit hypothesis [9]. Protein hyperactivation–related mechanisms [10] [11].

Among these concepts, the cholinergic hypothesis has been the subject of the most extensive experimental and clinical investigations [12]. Heterocyclic systems based on the pyrimidine scaffold and their annelated derivatives constitute a fundamentally important class of compounds in medicinal chemistry, natural product research, and synthetic organic chemistry [13]. The synthesis of various pyrido[2,3-*d*]pyrimidin-4-ones containing a halogen in the ring has been studied with great interest by several researchers [14]. Within this class, pyrido[2,3-*d*]pyrimidin-4-one derivatives are distinguished by their broad spectrum of biological activities, while rational structural modification provides access to new molecules with enhanced or altered pharmacological profiles [15]. In particular, the synthesis of halogen-substituted pyrido[2,3-*d*]pyrimidin-4-ones has been the subject of sustained research interest due to their unique reactivity and bioactivity patterns [14] [16]. Experimental studies have demonstrated that pyrido[2,3-d]pyrimidine frameworks may undergo alternative reaction pathways when exposed to oxidizing agents, halogenating reagents, or coordination-active species. From a synthetic standpoint, new derivatives can be efficiently obtained via high-temperature condensation reactions of 2-aminonicotinic acid with appropriate lactams, conducted either under solvent-free conditions or in organic solvents such as benzene, toluene, or 1,4-dioxane [13, 17-19].

Research methodology. In the first step, 2-amino-5-bromo-nicotinic acid (1) and the corresponding lactams were reacted in the presence of dichloroethane to synthesize pyrido[2,3-*d*]pyrimidinones (2a-c) (Scheme 1). The synthesized pyrido[2,3-*d*]pyrimidinones were then reacted with aromatic boronic acids in the presence of tetrakis(triphenylphosphine)palladium as a catalyst and sodium carbonate as a base, using a solvent mixture of 1,4-dioxane: water (3:2, v/v) at 100 °C for 12 hours. The reaction was monitored by TLC, and upon completion, the product was extracted into DCM. The organic phases were washed with saturated sodium chloride, followed by water (15×3 mL), and the solvent was removed under reduced pressure using a Rotavapor R-300. The reaction mixture was finally purified by column chromatography using a dichloromethane/methanol system (3a-c and 4a-c) (Scheme 2) [20].



1 **Scheme 1.** **2a-c** n = 1,2,3



2a-b **3a-c and 4a-c**

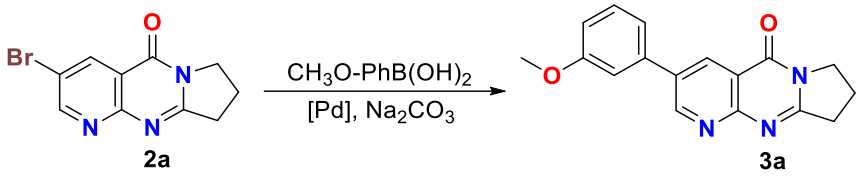
n=1: 3a-c, R = 3-OCH_3; R = 2-OCH_3; R = 4-$COCH_3$

n=2: 4a-c, R = 3-NH_2; R = 4-OH; R = 4-morpholine

Scheme 2

Table 1 presents the results of optimization of conditions for the Suzuki–Miyaura coupling reaction. The reaction was tested in the presence of a palladium catalyst, various bases, and a mixture of solvents [20, 21].

Table 1. Optimization of conditions for the Suzuki coupling reaction

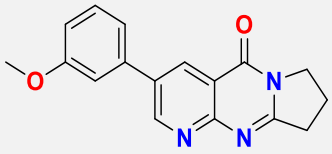
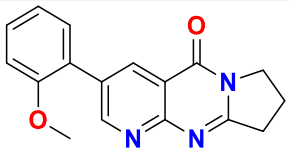
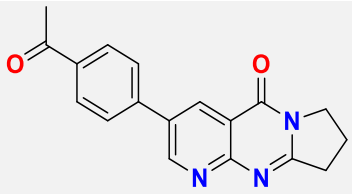
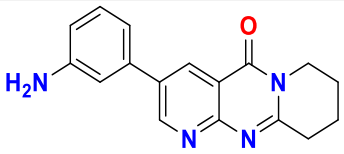
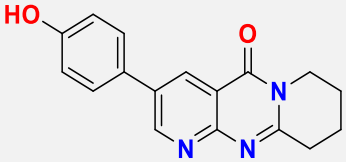
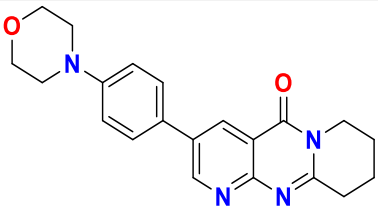


2a 3a

№	Catalyst	Base	Solvent	Temperature	Time	Yield(%)
3a	$Pd(PPh_3)_4$	Na_2CO_3	1,4-dioxane/water 3:2	100 °C	12 h	88
3a	$Pd(PPh_3)_4$	K_2CO_3	1,4-dioxane/water 5:2	100 °C	12 h	64
3a	$Pd(PPh_3)_4$	Na_2CO_3	1,4-dioxane/water 1:1	100 °C	12 h	54
3a	$Pd_2(dba)_3$	Cs_2CO_3	1,4-dioxane/water 3:2	100 °C	12 h	61

The table below presents the main physicochemical parameters of the synthesized compounds 3a–c, namely, yields, thin-layer chromatography Rf values, and melting points (MP) Table 2.

Table 2. Physical quantities of the synthesized compounds

№	Structure	Yield	R_f	Melting point
1.	**3a** 	88	DCM/MeOH=20:1, Rf=0.4	209-210 °C
2.	**3b** 	67	DCM/MeOH=20:1, Rf=0.37	130-131 °C
3.	**3c** 	37	DCM/MeOH=20:1, Rf=0.29	250-251 °C
4.	**4a** 	25	DCM/MeOH=20:1, Rf=0.21	223-224 °C
5.	**4b** 	63	DCM/MeOH=20:1, Rf=0.29	278-279 °C
6.	**4c** 	55	DCM/MeOH=20:1, Rf=0.31	253-254 °C

Analysis results.

3-methoxyphenyl)-8,9-dihydropyrido[2,3-*d*]pyrrolo[1,2-a]pyrimidin-5(7H)-one (3a). Yield 88 %, white solid, m.p: 209-210 °C, Rf=0.4, (DCM/Met=20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.12 (d, J = 2.6 Hz, 1H), 8.68 (d, J = 2.6 Hz, 1H), 7.35 (t, J = 7.9 Hz, 1H), 7.20 (d, J = 7.7 Hz, 1H), 7.13 (t, J = 2.1 Hz, 1H), 6.91 (dd, J = 8.2, 2.5 Hz, 1H), 4.17 (t, 2H), 3.82 (s, 3H), 3.21 (t, J = 8.0 Hz,

2H), 2.27 (dt, J = 14.8, 7.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.15, 161.40, 160.50, 158.61, 154.74, 138.08, 134.79, 133.48, 130.53, 119.77, 115.41, 114.29, 112.88, 55.64, 46.95, 32.98, 19.54. HRMS (ESI) calcd for $C_{17}H_{15}N_3O_2$ $[M+H]^+$ 294.1237 found 294.1233.

3-(2-methoxyphenyl)-8,9-dihydropyrido[2,3-*d*]pyrrolo[1,2-a]pyrimidin-5(7H)-one (3b). Yield 67 %, white solid, m.p: 130-131 ℃, Rf=0.37, (DCM/Met=20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.06 (d, *J* = 2.5 Hz, 1H), 8.66 (d, *J* = 2.5 Hz, 1H), 7.32 (t, *J* = 6.9 Hz, 2H), 7.01 (t, *J* = 7.5 Hz, 1H), 6.95 (d, *J* = 9.1 Hz, 1H), 4.16 (t, 2H), 3.77 (s, 3H), 3.20 (t, *J* = 8.0 Hz, 2H), 2.26 (dt, *J* = 14.8, 7.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.88, 161.47, 158.01, 156.95, 156.80, 135.89, 132.74, 130.90, 130.19, 125.98, 121.37, 115.08, 111.50, 55.74, 46.88, 32.93, 19.56. HRMS (ESI) calcd for $C_{17}H_{15}N_3O_2$ $[M+H]^+$ 294.1237 found 294.1232.

3-(4-acetylphenyl)-8,9-dihydropyrido[2,3-*d*]pyrrolo[1,2-a]pyrimidin-5(7H)-one (3c). Yield 37 %, yellow solid, m.p: 250-251 ℃, Rf=0.29, (DCM/Met=20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.16 (d, *J* = 2.7 Hz, 1H), 8.73 (d, *J* = 2.6 Hz, 1H), 8.02 (d, *J* = 8.1 Hz, 2H), 7.72 (d, *J* = 8.1 Hz, 2H), 4.19 (t, *J* = 7.3 Hz, 2H), 3.22 (t, *J* = 8.1 Hz, 2H), 2.59 (s, 3H), 2.29 (p, *J* = 7.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.63, 163.76, 161.21, 159.03, 154.56, 141.06, 136.95, 133.90, 129.48, 128.22, 127.46, 115.51, 47.04, 29.88, 26.92, 19.51. HRMS (ESI) calcd for $C_{18}H_{15}N_3O_2$ $[M+H]^+$ 306.1237 found 306.1233.

3-(3-aminophenyl)-7,8,9,10-tetrahydro-5H-dipyrido[1,2-a:2',3'-*d*]pyrimidin-5-one (4a). Yield 25 %, yellow solid, m.p: 223-224 ℃, Rf=0.21, (DCM: Met=20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.10 (d, *J* = 2.6 Hz, 1H), 8.64 (d, *J* = 2.6 Hz, 1H), 7.22 (d, *J* = 7.8 Hz, 1H), 7.00 (d, *J* = 8.3 Hz, 1H), 6.92 (s, 1H), 6.68 (d, *J* = 7.8 Hz, 1H), 4.02 (t, *J* = 6.2 Hz, 2H), 3.73 (s, 2H), 3.04 (t, *J* = 6.6 Hz, 2H), 1.95 (dt, *J* = 28.4, 6.6 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.98, 158.54, 156.65, 155.09, 147.41, 137.87, 135.03, 133.48, 130.41, 117.56, 115.30, 115.17, 113.69, 42.99, 32.44, 22.20, 19.41. HRMS (ESI) calcd for $C_{17}H_{16}N_4O$ $[M+H]^+$ 293.1397 found 293.1393.

3-(4-hydroxyphenyl)-7,8,9,10-tetrahydro-5H-dipyrido[1,2-a:2',3'-*d*]pyrimidin-5-one (4b). Yield 63 %, yellow solid, m.p: 278-279 ℃, Rf=0.29, (DCM/Met=20:1). ^{1}H NMR (400 MHz, PYRIDINE) δ 9.49 (d, *J* = 2.6 Hz, 1H), 8.91 (d, *J* = 2.7 Hz, 1H), 7.72 (d, *J* = 7.5 Hz, 2H), 7.32 (d, *J* = 7.3 Hz, 2H), 3.99 (t, *J* = 6.2 Hz, 2H), 3.62 (s, 1H), 2.98 (t, *J* = 6.3 Hz, 2H), 1.69 (dp, *J* = 25.3, 6.0 Hz, 4H). ^{13}C NMR (100 MHz, PYRIDINE) δ 163.26, 160.09, 158.50, 157.16, 154.89, 134.95, 132.57, 129.31, 128.39, 117.65, 116.26, 43.28, 32.84, 22.43, 19.82. HRMS (ESI) calcd for $C_{17}H_{15}N_3O_2$ $[M+H]^+$ 294.1237 found 294.1233.

3-(4-morpholinophenyl)-7,8,9,10-tetrahydro-5*H*-dipyrido[1,2-a:2',3'-*d*]pyrimidin-5-one (4c). Yield 55 %, yellow solid, m.p: 253-254 ℃, Rf=0.31, (DCM/Met=20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.11 (d, *J* = 2.7 Hz, 1H), 8.62 (d, *J* = 2.5 Hz, 1H), 7.56 (d, *J* = 8.7 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 4.02 (t, *J* = 5.8 Hz, 2H), 3.88 – 3.78 (m, 4H), 3.23 – 3.14 (m, 4H), 3.03 (t, *J* = 6.6 Hz, 2H), 2.05 – 1.86 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.01, 158.06, 156.07, 154.58, 151.49, 134.51, 132.26, 128.02, 127.70, 115.98, 115.24, 66.97, 48.92, 42.94, 32.39, 22.20, 19.42. HRMS (ESI) calcd for $C_{21}H_{22}N_4O_2$ $[M+H]^+$ 363.1816 found 363.1809.

Conclusion. The cross-coupling reaction between pyrido[2,3-*d*]pyrimidin-4-ones and aromatic boronic acids was successfully carried out. As a result of this reaction, aromatic fragments were efficiently introduced into the pyrido[2,3-*d*]pyrimidin-4-one scaffold, leading to the synthesis of new substituted derivatives. The synthetic process was characterized by high selectivity and stability, indicating that the reaction conditions were appropriately optimized. The structures and purity of the obtained compounds were reliably confirmed by ^{1}H NMR and ^{13}C NMR spectroscopy, while their molecular masses and isotopic compositions were determined using high-resolution liquid chromatography–mass spectrometry (LC-QTOF-HRMS). In addition, melting points and crystallization properties were determined using MP-560.

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