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Dedicated to Full Member of the Russian Academy of Sciences B.A. Trofimov on the 65th Anniversary of His Birth

Synthesis of Functionalized 2-(2-Pyrrolyl)pyrazolo[1,5-*a*]pyrimidines

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Abstract—Reactions of 5-amino-3-(2-pyrrolyl)pyrazoles with β-dicarbonyl compounds (acetylacetone and ethyl acetoacetate) lead to formation of up to 90% of polysubstituted pyrazolo[1,5-*a*]pyrimidines.

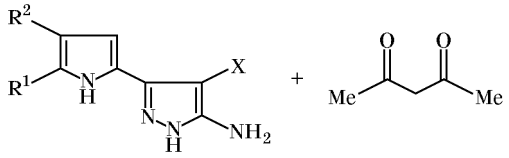
Reactions of aminopyrazoles with electrophilic reagents give rise to various fused heterocyclic compounds, including pyrazolo[1,5-*a*]pyrimidines which are synthetic analogs of purines. These compounds exhibit a wide spectrum of biological activity, in particular enzymatic [4], antibacterial [5], antiphlogistic [6], and antiparasitic [7]. Mustazza *et al.* [8] recently reported on the synthesis of new compounds of the pyrazolo[1,5-*a*]pyrimidine series, which are promising for the treatment of ahypnia.

The above data indicate that development of new synthetic approaches to pyrazolo[1,5-*a*]pyrimidines remains an important problem. The most extensively developed route to pyrazolo[1,5-*a*]pyrimidines is based on the reaction of aminopyrazoles with 1,3-dicarbonyl compounds [9, 10]. Also, syntheses with kethene dithioacetals [5] and carboxylic acid esters [11] have been reported.

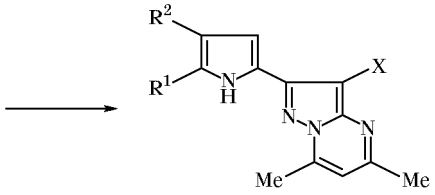
However, the wide series of known pyrazolo[1,5-*a*]pyrimidines includes no compounds having a pyrrole ring and functional groups which could be subjected to further modification. Related planar fused heterocyclic compounds capable of participating in charge transfer processes, hydrogen bond formation, and interplane self-assembling via π,π interaction between the rings attract great interest as potential DNA-interactive reagents.

With the goal of obtaining compounds possessing the above properties, we examined the condensation of 5-amino-3-(2-pyrrolyl)pyrazoles **Ia**–**If**, which are readily available from functionalized 2-vinylpyrroles [12–16] and hydrazine hydrate [14, 15, 17, 18], with dicarbonyl compounds, acetylacetone and ethyl acetoacetate. As a result, we isolated previously unknown pyrazolo[1,5-*a*]pyrimidines **IIa**–**IIf** (Scheme 1) and **IIIa**–**IIId** (Scheme 2) in 28–90% yield.

Scheme 1.



Ia–**If**



IIa–**IIf**

I, **II**, R^1 = Pr, R^2 = Et, X = $CONH_2$ (**a**); R^1 = Bu, R^2 = Pr, X = $CONH_2$ (**b**); R^1R^2 = $(CH_2)_4$, X = CN (**c**); R^1R^2 = $(CH_2)_4$, X = $CONH_2$ (**d**); R^1 = Ph, R^2 = H, X = CN (**e**); R^1 = Ph, R^2 = H, X = $CONH_2$ (**f**).

The reaction of pyrazole **Ie** with acetylacetone under the conditions given in [19] for the synthesis of 5,7-dimethyl-2-phenylpyrazolo[1,5-*a*]pyrimidine (ethanol containing a catalytic amount of acetic acid; 1 h at room temperature) afforded no expected pyrazolopyrimidine **IIe**. We succeeded in obtaining

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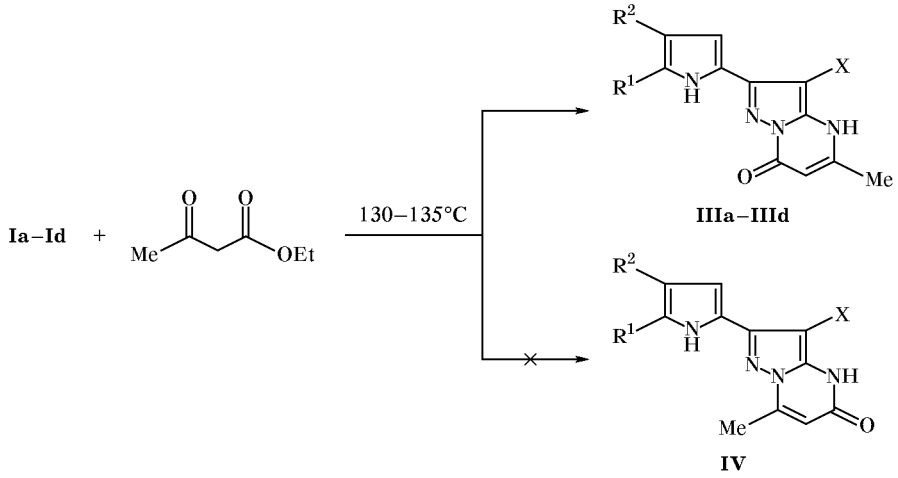
Scheme 2.



III, R^1 = Pr, R^2 = Et, X = $CONH_2$ (**a**); R^1 = Bu, R^2 = Pr, X = $CONH_2$ (**b**); R^1R^2 = $(CH_2)_4$, X = CN (**c**); R^1R^2 = $(CH_2)_4$, X = $CONH_2$ (**d**).

compound **IIe** in as poor as 28% yield only in 72 h. Under analogous conditions, from aminopyrazole **Ic** we obtained 38% of pyrazolopyrimidine **IIc**. The yield of product **IIc** considerably increased (to 74%) when initial pyrazole **Ic** was heated for 2 h in boiling ethanol with 2 equiv of acetylacetone. In this way, pyrazolo[1,5-*a*]pyrimidines **IIa** and **IIb** were synthesized in 89 and 62% yield, respectively, from the corresponding pyrazoles **Ia** and **Ib**. However, pyrazoles **Id** and **If** failed to react with acetylacetone even under these conditions. Presumably, the reason is their poor solubility in ethanol and hence low concentration in the reaction solution. Pyrazolo[1,5-*a*]pyrimidines **IId** and **IIf** were synthesized in 88 and 90% yield, respectively, when the reaction was performed without a solvent at elevated temperature (130–155°C, 2 h).

Heating of aminopyrazoles **Ia**–**Id** with ethyl acetoacetate for 7 h at 130–135°C resulted in selective formation of pyrazolo[1,5-*a*]pyrimidines **IIIa**–**IIId** (yield 65–73%, Scheme 2).

The ^{1}H NMR spectra give no reliable information on the structure of products **III**; on the other hand, they indicate that only one of possible isomeric products **III** and **IV** is formed rather than their mixture. Taking into account the higher nucleophilicity of the primary amino group relative to the endocyclic pyrazole nitrogen atom and unambiguously proved structure of pyrazolo[1,5-*a*]pyrimidines synthesized from aminopyrazoles and ethyl acetoacetate (see [10]), the condensation products were assigned structure **III**.

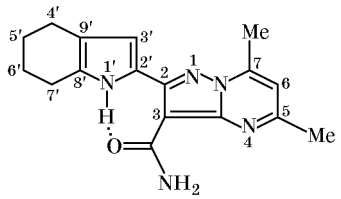
Pyrazolopyrimidines **IIa**–**IIf** and **IIIa**–**IIId** are high-melting crystalline substances which are poorly soluble in most organic solvents (except for DMSO). The structure of products **IIa**–**IIf** and **IIIa**–**IIId** was confirmed by IR and ^{1}H and ^{13}C NMR spectroscopy. Their IR spectra contain absorption bands typical of NH stretching vibrations as sharp peaks on the background of a diffuse absorption band in the region 3155–3445 cm^{-1}. Insofar as molecules **IIa**, **IIb**, **IId**, **IIf**, **IIIa**, **IIIb**, and **IIId** possess NH groups belonging to the pyrrole, carbamoyl, and (compounds **IIIa**–**IIId**) pyrimidine fragments, accurate assignment of the NH bands in the spectra of crystalline samples is hardly possible. The amide carbonyl group gives rise to absorption at 1645–1669 cm^{-1} (amide II band), and stretching vibrations of the C≡N bonds in compounds **IIc**, **IIe**, and **IIIc** appear at 2215–2220 cm^{-1}. Strong absorption bands at 1605–1628 cm^{-1} belong to vibrations of the pyrimidine ring. Carbonyl absorption bands of compounds **IIIa**–**IIId** are located in the region 1687–1702 cm^{-1}.

The ^{1}H and ^{13}C signals in the NMR spectra of pyrazolopyrimidines **IIa**–**IIf** were assigned on the basis of the results of two-dimensional NOESY, HSQC, and HMBC experiments [20]. For example, a broadened singlet at δ 12.65 ppm in the spectrum of **IId** was assigned to the NH proton by the double resonance technique. Suppression of that signal leads to disappearance of splitting of the 3′-H signal ($^4J_{NH,3'-H}$ = 1.9 Hz). In addition, a correlation was observed in the 2M NOESY spectrum of **IId** between the NH proton and methylene protons on $C^{7'}$. The downfield shift of the NH signal to a region which is not typical of 4,5,6,7-tetrahydroindole [21] is explained by formation of intramolecular hydrogen bond between the NH proton and carbonyl oxygen

Table 1. Yields, melting points, and elemental analyses of pyrazolopyrimidines **IIa**–**IIf** and **IIIa**–**IIId**

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
IIa	89	252	65.22	7.99	21.51	$C_{18}H_{23}N_5O$	66.44	7.12	21.52
IIb	62	230	68.75	7.79	19.61	$C_{20}H_{27}N_5O$	67.96	7.70	19.81
IIc	74	>300	69.51	6.32	22.49	$C_{17}H_{17}N_5$	70.08	5.88	24.04
IId	88	290	65.80	6.30	22.62	$C_{17}H_{19}N_5O$	66.00	6.19	22.64
IIe	28	>300	70.79	5.78	22.20	$C_{19}H_{15}N_5$	72.83	4.82	22.35
IIf	90	>300	67.56	5.21	19.62	$C_{19}H_{17}N_5O$	68.87	5.17	21.13
IIIa	65	286	62.33	6.14	21.70	$C_{17}H_{21}N_5O_2$	62.31	6.47	21.39
IIIb	71	288	63.99	7.15	19.36	$C_{19}H_{25}N_5O_2$	64.20	7.09	19.70
IIIc	65	>300	65.47	5.27	23.97	$C_{16}H_{15}N_5O$	65.52	5.15	23.88
IIId	73	>300	61.50	5.60	22.13	$C_{16}H_{17}N_5O_2$	61.71	5.50	22.49

atom. Such intramolecular hydrogen bond can be formed when the molecular fragments are arranged as shown below:



IId

Protons of the carbamoyl group in **IId** give rise to two signals at δ 8.99–9.10 and 5.57–5.69 ppm, presumably due to the presence of two conformers arising from restricted rotation about the N–C=O bond. The methyl proton signals were assigned by analysis of correlations observed in the 2M HMBC spectrum. The downfield singlet at δ 2.75 ppm gives a cross peak with the ^{13}C resonance at δ_C 147.29 ppm (C^7). The singlet at δ 2.57 ppm is coupled with the ^{13}C signal at δ_C 160.93 ppm (C^5). Both methyl group signals show a correlation with the ^{13}C signal at δ_C 110.97 ppm (C^6), and the 6-H signal shows cross peaks due to couplings through three bonds with both ^{13}C signals from the methyl groups at δ_C 18.46 and 25.94 ppm. These data support the assumed structure of the given fragment. Resonance signals from the quaternary $C^{8'}$ and $C^{9'}$ carbon nuclei were assigned on the basis of correlations with protons in the fused cyclohexene ring.

Thus we were the first to synthesize a series of functionalized pyrazolo[1,5-*a*]pyrimidine derivatives having 2-pyrrolyl fragments. These compounds are promising for further modifications at the functional groups and as potential biologically active substances.

EXPERIMENTAL

The IR spectra were recorded on a Bruker IFS-25 spectrometer in KBr. The ^1H and ^{13}C NMR spectra were obtained on a Bruker DPX-250 instrument at 250.13 and 62.9 MHz, respectively, from solutions in $CDCl_3$ (**IIa**–**IIf**) and DMSO-d_6 (**IIIa**–**IIId**); HMDS was used as internal reference. Initial 5-aminopyrazoles **Ia**–**If** were synthesized by the procedures reported in [17, 18].

2-(4-Ethyl-5-propyl-2-pyrrolyl)-5,7-dimethylpyrazolo[1,5-*a*]pyrimidine-3-carboxamide (IIa). A mixture of 0.105 g (0.40 mmol) of 5-aminopyrazole **Ia**, 0.080 g (0.80 mmol) of acetylacetone, and 2 drops of glacial acetic acid in 6 ml of anhydrous alcohol was heated for 2 h under reflux. The mixture was cooled, and light yellow fine crystals were filtered off, washed with diethyl ether (2 × 3 ml), and dried. Yield 0.116 g (89%).

2-(5-Butyl-4-propyl-2-pyrrolyl)-5,7-dimethylpyrazolo[1,5-*a*]pyrimidine-3-carboxamide (IIb). Following the above procedure, from 0.106 g (0.37 mmol) of 5-aminopyrazole (**Ib**) and 0.070 g (0.74 mmol) of acetylacetone we obtained 0.080 g (62%) of compound **IIb** as light yellow crystals.

5,7-Dimethyl-2-(4,5,6,7-tetrahydroindol-2-yl)pyrazolo[1,5-*a*]pyrimidine-3-carbonitrile (IIc). A mixture of 0.500 g (2.20 mmol) of 5-aminopyrazole **Ic**, 0.440 g (4.40 mmol) of acetylacetone, and 2 drops of glacial acetic acid in 90 ml of anhydrous alcohol was heated for 2 h under reflux. The mixture was cooled, and brown fine needle-like crystals were filtered off, washed with diethyl ether (3 × 5 ml), and dried. Yield 0.408 g (64%). The filtrate was partially evaporated to isolate an additional portion of the product, 0.066 g (10%).

Table 2. ^{1}H NMR and IR spectra of pyrazolopyrimidines **IIa**–**IIf** and **IIIa**–**IIId**

Comp. no.	^{1}H NMR spectrum, δ, ppm								IR spectrum, ν, cm^{-1}
	R^1	R^2	3′-H	NH	X	6-H	5-Me	7-Me	
IIa	2.47, 1.72, 0.99	2.62, 1.21	6.93	12.84	9.01, 5.57	6.63	2.59	2.78	490, 524, 556, 637, 714, 792, 821, 842, 1032, 1062, 1160, 1180, 1221, 1275, 1294, 1328, 1340, 1372, 1390, 1419, 1448, 1462, 1513, 1553, 1620, 1645, 2868, 2927, 2956, 3055, 3200, 3297, 3427
IIb	2.32, 1.64, 1.40, 0.94	2.64, 1.64, 0.94	6.91	12.78	9.02, 5.62	6.62	2.58	2.77	525, 637, 714, 792, 822, 841, 1034, 1180, 1219, 1252, 1276, 1307, 1338, 1372, 1391, 1418, 1447, 1464, 1512, 1553, 1620, 1650, 2865, 2926, 2958, 3057, 3286, 3430
IIc	2.64, 1.84,	2.57, 1.77	6.96	8.68	–	6.67	2.57	2.71	495, 520, 545, 564, 630, 708, 729, 761, 810, 833, 926, 947, 1026, 1050, 1127, 1146, 1161, 1195, 1230, 1270, 1296, 1355, 1371, 1388, 1437, 1526, 1558, 1605, 1620, 2220, 2852, 2929, 3006, 3206, 3297, 3455
IId	2.70, 1.82,	2.57, 1.76	6.85	12.64	8.99, 5.57	6.62	2.57	2.76	527, 555, 579, 635, 703, 723, 795, 816, 828, 1043, 1108, 1134, 1156, 1180, 1216, 1272, 1354, 1377, 1424, 1443, 1463, 1515, 1552, 1615, 1648, 2825, 2850, 2920, 3071, 3195, 3275, 3372
IIe	7.64, 7.45, 7.31	6.65	7.26	9.35	–	6.65	2.62	2.78	518, 546, 564, 634, 656, 668, 682, 718, 750, 760, 795, 840, 897, 925, 943, 1027, 1054, 1073, 1158, 1173, 1229, 1249, 1290, 1370, 1382, 1406, 1418, 1439, 1485, 1536, 1555, 1578, 1600, 1621, 2215, 3036, 3061, 3267, 3308, 3461
IIf	7.69, 7.38, 7.37	6.66	7.15	13.97	9.10, 5.69	6.70	2.62	2.81	533, 610, 623, 644, 678, 690, 708, 721, 746, 772, 793, 827, 843, 904, 925, 1027, 1049, 1078, 1160, 1172, 1205, 1230, 1269, 1280, 1316, 1349, 1374, 1393, 1405, 1426, 1442, 1466, 1476, 1527, 1549, 1577, 1614, 1647, 3073, 3185, 3342
IIIa[a]	2.47, 1.56, 1.11	2.47, 1.21	6.46	11.19	7.28	5.67	2.34	–	542, 577, 655, 711, 737, 755, 806, 835, 1041, 1148, 1180, 1270, 1309, 1328, 1371, 1396, 1435, 1494, 1518, 1587, 1662, 1697, 2870, 2930, 2959, 3188, 3228, 3295, 3431
IIIb[a]	2.64, 1.64, 1.13	2.64, 0.94	6.48	11.20	7.27	5.69	2.32	–	445, 581, 658, 713, 726, 757, 810, 945, 1022, 1041, 1158, 1179, 1256, 1276, 1326, 1370, 1399, 1433, 1488, 1519, 1587, 1612, 1663, 1687, 2860, 2927, 2955, 3183, 3313, 3372
IIIc[a]	2.49,	1.70	6.61	11.28	–	5.71	2.31	–	443, 517, 579, 693, 712, 721, 738, 805, 825, 929, 1025, 1043, 1057, 1134, 1161, 1197, 1221, 1272, 1300, 1335, 1356, 1369, 1406, 1440, 1474, 1531, 1584, 1605, 1628, 1669, 2219, 2831, 2932, 2993, 3079, 3155, 3423
IIId[a]	2.59,	1.70	6.42	11.65	7.38	5.69	2.35	–	455, 530, 546, 650, 697, 710, 778, 790, 815, 840, 928, 1056, 1133, 1179, 1226, 1272, 1352, 1415, 1499, 1522, 1576, 1620, 1652, 1702, 2847, 2931, 3179, 3312, 3467

[a] Signals from the NH protons of the pyrimidine ring in compounds **IIIa**–**IIId** are located at δ 12.31–13.10 ppm.

5,7-Dimethyl-2-(4,5,6,7-tetrahydroindol-2-yl)-pyrazolo[1,5-*a*]pyrimidine-3-carboxamide (IId). *a.* A mixture of 0.095 g (0.39 mmol) of 5-aminopyrazole **Id** and 0.970 g (1 ml, 9.70 mmol) of acetylacetone was heated for 2 h at 130–135°C. After cooling, the precipitate was filtered off, washed with diethyl ether (3 × 5 ml), and dried. Yellow crystals. Yield 0.105 g (87%).

b. A mixture of 0.190 g (0.78 mmol) of pyrrolopyrazole (**Id**) and 0.970 g (1 ml, 9.70 mmol) of acetylacetone was heated for 2 h at 150–155°C. Yield 0.210 g (88%).

5,7-Dimethyl-2-(5-phenyl-2-pyrrolyl)pyrazolo-[1,5-*a*]pyrimidine-3-carbonitrile (IIe). Acetylacetone, 0.050 g (0.05 mmol), was added to a solution of 0.128 g (0.50 mmol) of 5-aminopyrazole **Ie** in 10 ml of anhydrous alcohol, and a drop of glacial acetic acid was added. The mixture was left to stand for 72 h at room temperature, and the brown crystals were filtered off, washed with diethyl ether (3 × 3 ml), and dried. Yield 0.041 g (28%).

5,7-Dimethyl-2-(5-phenyl-2-pyrrolyl)pyrazolo-[1,5-*a*]pyrimidine-3-carboxamide (IIf). *a.* A mixture of 0.082 g (0.31 mmol) of 5-aminopyrazole **If** and 0.970 g (1 ml, 9.70 mmol) of acetylacetone was heated for 2 h at 130–135°C. After cooling, the crystals were filtered off, washed with ether (3 × 5 ml), and dried. Yellow crystals. Yield 0.080 g (78%).

b. A mixture of 0.200 g (0.75 mmol) of 5-aminopyrazole **If** and 0.970 g (1 ml, 9.70 mmol) of acetylacetone was heated for 2 h at 150–155°C. Yield of **IIf** 0.224 g (90%).

2-(4-Ethyl-5-propyl-2-pyrrolyl)-5-methyl-7-oxo-4,7-dihydropyrazolo[1,5-*a*]pyrimidine-3-carboxamide (IIIa). A mixture of 0.200 g (0.76 mmol) of 5-aminopyrazole **Ia** and 5 ml of ethyl acetoacetate was heated for 7 h at 130–135°C. The mixture was cooled, 20 ml of diethyl ether was added, and the light brown crystals were filtered off, washed with ethanol, and dried. Yield 0.162 g (65%).

2-(5-Butyl-4-propyl-2-pyrrolyl)-5-methyl-7-oxo-4,7-dihydropyrazolo[1,5-*a*]pyrimidine-3-carboxamide (IIIb). Following the above procedure, from 0.200 g (0.69 mmol) of 5-aminopyrazole **Ib** we obtained 0.174 g (71%) of compound **IIIb** as light brown crystals.

5-Methyl-7-oxo-2-(4,5,6,7-tetrahydroindol-2-yl)-4,7-dihydropyrazolo[1,5-*a*]pyrimidine-3-carbonitrile (IIIc). Following the above procedure, from 0.200 g (0.88 mmol) of 5-aminopyrazole **Ic** we obtained 0.167 g (65%) of compound **IIIc** as light brown crystals.

5-Methyl-7-oxo-2-(4,5,6,7-tetrahydroindol-2-yl)-4,7-dihydropyrazolo[1,5-*a*]pyrimidine-3-carboxamide (IIId). Likewise, from 0.200 g (0.82 mmol) of 5-aminopyrazole **Id** we obtained 0.185 g (73%) of compound **IIId** as light brown crystals.

The yields, melting points, and elemental analyses of compounds **IIa**–**IIf** and **IIIa**–**IIId** are given in Table 1, and Table 2 contains their ^{1}H NMR and IR spectral parameters.

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