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Synthesis of *N*-substituted 2-aminomethyl-5-methyl-7-phenyloxazolo[5,4-*b*]pyridines

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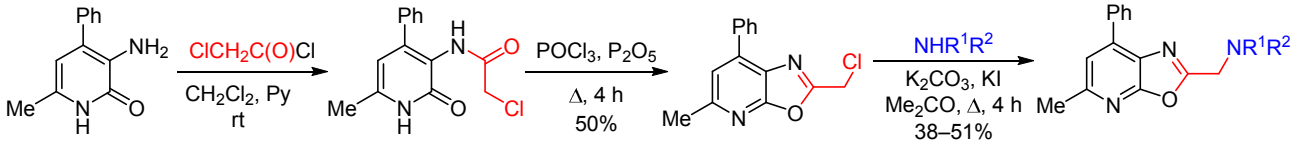


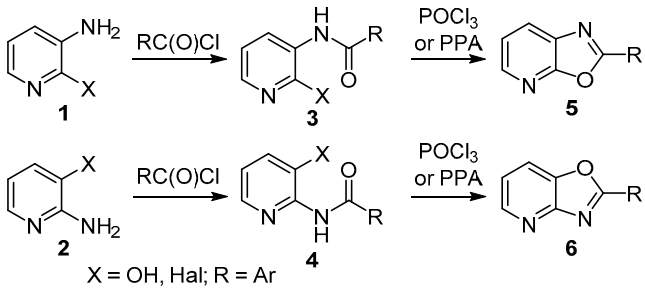
2-(Chloromethyl)-5-methyl-7-phenyloxazolo[5,4-*b*]pyridine was obtained by intramolecular cyclization based on the previously synthesized 2-chloro-*N*-(6-methyl-2-oxo-4-phenyl-1,2-dihydropyridin-3-yl)acetamide. The possibility of its use in the synthesis of previously unknown 2-aminomethyloxazolo[5,4-*b*]pyridine derivatives by nucleophilic substitution with various amines and a cyclic amide has been shown.

Keywords: 2-aminomethyloxazolo[5,4-*b*]pyridine derivatives, 3-amino-4-phenylpyridin-2(1*H*)-one, chloroacetamide, oxazolo[5,4-*b*]-pyridine, intramolecular cyclization, nucleophilic substitution.

Derivatives of oxazolo[5,4-*b*]pyridine and oxazolo[4,5-*b*]-pyridine derivatives are known to exhibit antimicrobial,[1] antitumor,[2] pronounced anti-inflammatory,[3] and analgesic activity.[4] Anti-inflammatory activity of some of them, for example, is comparable to that of phenylbutazone or indomethacin, while at the same time they do not cause irritation of the mucous membrane of the gastrointestinal tract, which can result from taking acidic anti-inflammatory drugs.[3]

There are several synthetic routes to access 2-substituted oxazolo[5,4-*b*]- and oxazolo[4,5-*b*]pyridines.[5–10] To date, the most common method is to use 3-amino-2-hydroxy- or 3-amino-2-halopyridines **1** or 2-amino-3-hydroxy- or 2-amino-3-halopyridines **2** as precursors, which, after forming the corresponding benzamides **3**, **4**, cyclize into the corresponding oxazolopyridines **5**, **6** in acidic media, for example, in the presence of polyphosphoric acid or phosphorus oxychloride (Scheme 1).

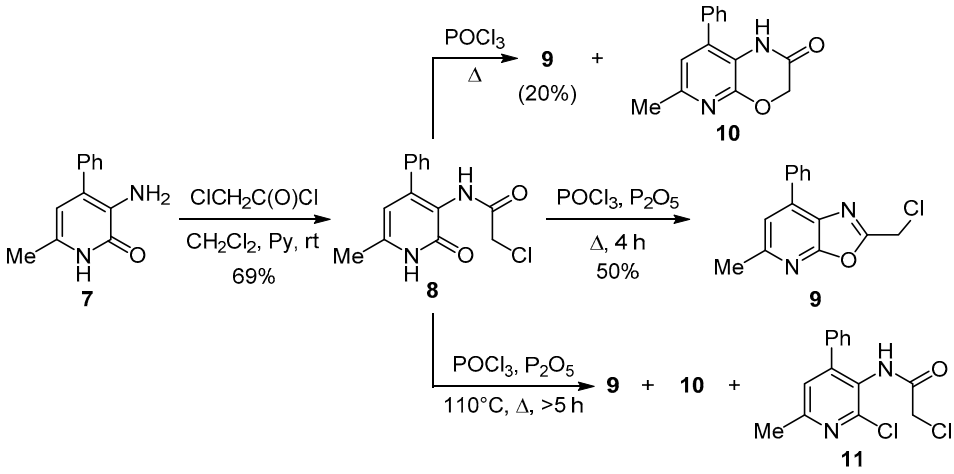
Scheme 1



It should also be noted that oxazolo[5,4-*b*]pyridine derivatives **5**, derived from *N*-(2-hydroxypyridin-3-yl)-benzamides **3** with unsubstituted pyridine ring, that is, amides of aromatic, and not aliphatic acids are widely represented in the literature. 5,7-Disubstituted and 2-alkyl-substituted oxazolo[5,4-*b*]pyridines are presented in the literature only by a few examples.[10,11] The corresponding 4,6-disubstituted 3-aminopyridin-2(1*H*)-ones have not been used for the synthesis of oxazolo[5,4-*b*]pyridines **5**.

As part of an ongoing study on the synthesis of 3-aminopyridin-2(1*H*)-one derivatives,[12–17] it was of interest to synthesize oxazolo[5,4-*b*]pyridine **9** from

Scheme 2



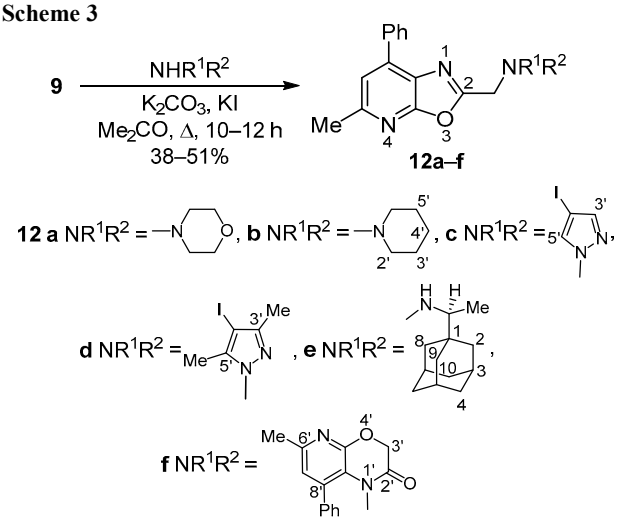
chloroacetamide **8**, which we previously obtained by acylation of 3-amino-6-methyl-4-phenylpyridin-2(1*H*)-one (**7**),[17] and to study the possibility of using compound **9** in the synthesis of 2-aminomethyl derivatives of oxazolo[5,4-*b*]-pyridines which are not described in literature (Scheme 2).

Cyclization of chloroacetamide **8** by heating with $POCl_3$ using the well-known classical procedure for the synthesis of oxazolo[5,4-*b*]pyridines **3** allowed us to obtain oxazolopyridine **9** with a rather low yield of about 20%. The reaction was complicated by the formation of by-products. When analyzing the chromato-mass spectra of the crude reaction product, we fixed 1*H*-pyrido[2,3-*b*][1,4]-oxazin-2(3*H*)-one **10**, resulting from the cyclization of compound **8**. Earlier, we already described the preparation of this compound in basic media.[17]

In order to increase the yield of oxazolo[5,4-*b*]pyridine **9**, we optimized the reaction conditions, which consisted in varying the excess of $POCl_3$ and the reaction time. The best yield of 2-(chloromethyl)-5-methyl-7-phenyloxazolo[5,4-*b*]-pyridine (**9**) (50%) was obtained by heating under reflux with a tenfold excess of $POCl_3$ with the addition of a threefold excess of P_2O_5 for 4 h. Longer heating resulted in increasing formation of a difficult to separate mixture of products, the ^{1}H NMR spectra of which revealed, in addition to signals from protons of compounds **9**, **10**, a distinct singlet at 8.11 ppm, most likely belonging to the proton H-5 of the formed 2-chloropyridine derivative **11** (Scheme 2).[18] The resulting 2-(chloromethyl)-5-methyl-7-phenyloxazolo[5,4-*b*]pyridine (**9**) was isolated in the form of light-beige crystals, easily soluble in almost all organic solvents, except hydrocarbons.

Next, we studied substitution of the chlorine atom in oxazolo[5,4-*b*]pyridine **9** by the amino group in reactions with morpholine, piperidine, 1-(adamantan-1-yl)ethan-1-amine (active ingredient of the antiviral drug rimantadine), 4-iodopyrazole, 4-iodo-3,5-dimethylpyrazole, and 1*H*-pyrido-[2,3-*b*][1,4]oxazin-2(3*H*)-one. Nucleophilic substitution was performed by heating in Me_2CO in the presence of K_2CO_3 and catalytic amounts of KI. The yields of products **12a**–**f** were in the range of 38–51% (Scheme 3). Although carrying the reaction in DMF significantly shortened the reaction time (from 10–12 to 3 h), it was accompanied by the formation of byproducts (according to TLC).

Scheme 3



Compounds **12a**,**c**–**f** represent beige or light-beige crystalline substances (compound **12b** is an oil), easily soluble in most organic solvents and partially in hexane when heated. The structures of the obtained compounds **12a**–**f** were confirmed by ^{1}H and ^{13}C NMR spectroscopy and mass spectrometry.

To conclude, we obtained for the first time the corresponding 2-(chloromethyl)-5-methyl-7-phenyloxazolo[5,4-*b*]-pyridine on the basis of 2-chloro-*N*-(6-methyl-2-oxo-4-phenyl-1,2-dihydropyridin-3-yl)acetamide. It can be used further in the nucleophilic substitution reaction for the synthesis of *N*-substituted 2-aminomethyloxazolo[5,4-*b*]-pyridine derivatives.

Experimental

IR spectra were registered on an FT-801 spectrometer in KBr pellets. ^{1}H and ^{13}C NMR spectra were acquired on a Jeol JNM-ECA 400 spectrometer (400 and 100 MHz, respectively) in $CDCl_3$, with TMS as internal standard or using residual solvent signals (7.26 ppm) to assign chemical shifts. Signals of ^{13}C atoms were assigned using the APT method, taking into consideration the known range of chemical shifts of carbon atoms in functional groups and those in the starting materials previously established by two-dimensional correlation spectra.[17] Mass spectra were recorded on an Agilent Technologies 5977E

(MSD) GC-MS, 7820A (GC System), helium carrier gas, carrier gas flow 0.9 ml/min, Agilent 1901S 433UI column: evaporator temperature 250°C, thermostat temperature 70–290°C, temperature gradient 10°C/min, EI ionization, 70 eV. Recording of the mass spectrum of compound **12f** as well as the determination of its molecular mass was performed on a Thermo Electron Double Focusing System high-resolution mass spectrometer (EI ionization, 70 eV) using perfluorokerosene (PFK) as the standard. Elemental analysis was performed on a Fisons EA 1106 Elemental analyzer. Melting points were determined on a Kofler bench. Monitoring of the reaction progress and assessment of the purity of synthesized compounds was done by TLC on Sorbfil AF-A-UV plates, visualization with UV light or in the iodine chamber.

The precursors 3-amino-6-methyl-4-phenylpyridin-2(1*H*)-one (**7**) and 2-chloro-*N*-(6-methyl-2-oxo-4-phenyl-1,2-dihydropyridin-3-yl)acetamide (**8**) were prepared following methods published by us previously.[17]

2-(Chloromethyl)-5-methyl-7-phenyloxazolo[5,4-*b*]pyridine (9). A mixture of 2-chloro-*N*-(6-methyl-2-oxo-4-phenyl-1,2-dihydropyridin-3-yl)acetamide (**8**) (277 mg, 1.0 mmol), $POCl_3$ (1.0 ml, 10 mmol), and P_2O_5 (426 mg, 3.0 mmol) was heated under reflux for 4 h. The excess $POCl_3$ was evaporated, and the thick residue was carefully treated with ice water. The formed precipitate was thoroughly triturated, filtered, washed with water, and air-dried. It was recrystallized from hexane–*i*-PrOH, 5:1 mixture. Yield 127 mg (50%), light-beige crystals, mp 126–128°C. IR spectrum, ν, cm^{-1}: 1622 (C=N), 2875 (C–H). ^{1}H NMR spectrum, δ, ppm: 2.68 (3H, s, CH_3); 4.74 (2H, s, CH_2Cl); 7.38 (1H, s, H-6); 7.45–7.53 (3H, m, H-3,4,5 Ph); 8.0 (2H, dd, ^3J = 8.3, ^4J = 1.5, H-2,6 Ph). ^{13}C NMR spectrum, δ, ppm: 24.4 (CH_3); 36.5 (CH_2Cl); 118.8 (C-6); 127.8; 128.8 (C-2,6 Ph); 128.9 (C-3,5 Ph); 129.7 (C-4 Ph); 134.4; 141.6; 155.9 (C-3a); 160.0 (C-2); 160.4 (C-5). Mass spectrum, *m*/*z* (I_{rel}, %): 260 $[M]^+$ (33), 258 $[M]^+$ (100), 218 (16), 154 (9), 127 (11). Found, %: C 64.66; H 4.41; N 10.99. $C_{14}H_{11}ClN_2O$. Calculated, %: C 65.00; H 4.29; N 10.83.

Synthesis of *N*-substituted 2-(aminomethyl)-5-methyl-7-phenyloxazolo[5,4-*b*]pyridines 12a–f (General method). A mixture of 2-(chloromethyl)-5-methyl-7-phenyloxazolo-[5,4-*b*]pyridine (**9**) (260 mg, 1.0 mmol) and the corresponding amine (1.1 mmol) was heated under reflux in Me_2CO (10 ml) in the presence of K_2CO_3 (276 mg, 2.0 mmol) (twofold excess of the amine was used for the synthesis of compounds **12a**,**b**) and a catalytic amount of KI (3 mg). The progress of the reaction was monitored by TLC, eluent hexane–$CHCl_3$–Me_2CO, 1:2:1. The reaction mixture was filtered from the precipitate of salts, washed with Me_2CO, and the solvent was evaporated. The residue was recrystallized from hexane–*i*PrOH, 2:1 mixture.

5-Methyl-2-(morpholin-4-ylmethyl)-7-phenyloxazolo-[5,4-*b*]pyridine (12a). Yield 118 mg (38%), light-beige crystals, mp 74–76°C. IR spectrum, ν, cm^{-1}: 1620 (C=N), 2960 (C–H). ^{1}H NMR spectrum, δ, ppm: 2.70–2.72 (7H, m, CH_3, $N(CH_2)_2$); 3.76 (4H, br. t, J = 4.6, $O(CH_2)_2$); 3.93 (2H, s, CH_2); 7.39 (1H, s, H-6); 7.47 (1H, t, J = 7.6, H-4 Ph); 7.52–7.55 (2H, m, H-3,5 Ph); 8.05 (2H, d, J = 7.6, H-2,6 Ph). ^{13}C NMR spectrum, δ, ppm.: 24.4 (CH_3); 53.2 ($N(CH_2)_2$); 55.6 (NCH_2); 66.8 ($O(CH_2)_2$); 118.5 (C-6); 127.7; 128.8 (C-2,6 Ph); 128.9 (C-3,5 Ph); 129.5 (C-4 Ph); 134.7; 141.0; 154.8 (C-3a); 160.4 (C-2); 162.0 (C-5). Mass spectrum, *m*/*z* (I_{rel}, %): 309 $[M]^+$ (0.5), 226 (16), 224 (100), 195 (4), 127 (5), 86 (7). Found, %: C 69.41; H 6.61; N 13.19. $C_{18}H_{19}N_3O_2$. Calculated, %: C 69.88; H 6.19; N 13.58.

5-Methyl-2-(piperidin-1-ylmethyl)-7-phenyloxazolo-[5,4-*b*]pyridine (12b). Yield 138 mg (45%), viscous oil. IR spectrum, ν, cm^{-1}: 1600 (C=N), 2853, 2960 (C-H). ^{1}H NMR spectrum, δ, ppm: 1.40–1.42 (2H, m, 4'-CH_2); 1.60–1.63 (4H, m, 3',5'-CH_2); 2.61 (4H, br. s, $N(CH_2)_2$); 2.68 (3H, s, CH_3); 3.90 (2H, s, CH_2); 7.36 (1H, s, H-6); 7.44 (1H, t, J = 7.6, H-4 Ph); 7.52 (2H, t, J = 6.9 H-3,5 Ph); 8.04 (2H, d, J = 7.6, H-2,6 Ph). ^{13}C NMR spectrum, δ, ppm: 23.8 (C-4'); 24.3 (CH_3); 25.9 (C-3',5'); 54.2 (C-2',6'); 56.0 (NCH_2); 118.3 (C-6); 127.8; 128.8 (C-2,6 Ph); 128.9 (C-3,5 Ph); 129.4 (C-4 Ph); 129.9; 134.8; 140.8; 154.5 (C-3a); 160.5 (C-2); 162.9 (C-5). Found, %: C 73.81; H 6.45; N 13.29. $C_{19}H_{21}N_3O$. Calculated, %: C 74.24; H 6.89; N 13.67.

2-[(4-Iodo-1*H*-pyrazol-1-yl)methyl]-5-methyl-7-phenyl-oxazolo[5,4-*b*]pyridine (12c). Yield 172 mg (40%), beige crystals, mp 145–148°C. IR spectrum, ν, cm^{-1}: 688 (C–I), 1616 (C=N), 2926 (C–H), 3051, 3125 (H–C=). ^{1}H NMR spectrum, δ, ppm: 2.69 (3H, s, CH_3); 5.62 (2H, s, CH_2); 7.41 (1H, s, H-6); 7.47–7.56 (3H, m, H-3,4,5 Ph); 7.57 (1H, s, H-3'); 7.71 (1H, s, H-5'); 8.04 (2H, d, J = 6.1, H-2,6 Ph). ^{13}C NMR spectrum, δ, ppm.: 24.4 (CH_3); 49.7 (CH_2); 57.7 (C–I); 118.8 (C-6); 127.6; 128.8 (C-2,6 Ph); 128.9 (C-3,5 Ph); 129.7 (C-4 Ph); 134.3; 134.5 (C-5'); 141.7; 145.5 (C-3'); 155.7; 158.6 (C-2); 160.3 (C-5). Found, %: C 49.44; H 3.41; N 13.22. $C_{17}H_{13}IN_4O$. Calculated, %: C 49.06; H 3.18; N 13.46.

2-[(4-Iodo-3,5-dimethyl-1*H*-pyrazol-1-yl)methyl]-5-methyl-7-phenyloxazolo[5,4-*b*]pyridine (12d). Yield 206 mg (43%), beige crystals, mp 160–163°C. IR spectrum, ν, cm^{-1}: 624 (C–I), 1620 (C=N), 1638 (C=N), 2924 (C–H). ^{1}H NMR spectrum, δ, ppm: 2.19 (3H, s, 3'-CH_3); 2.41 (3H, s, 5'-CH_3); 2.66 (3H, s, CH_3); 5.52 (2H, s, CH_2); 7.38 (1H, s, H-6); 7.43–7.53 (3H, m, H-3,4,5 Ph); 8.03 (2H, d, J = 7.6, H-2,6 Ph). ^{13}C NMR spectrum, δ, ppm: 12.2 (3'-CH_3); 14.1 (5'-CH_3); 24.3 (CH_3); 47.7 (CH_2); 64.2 (C–I); 118.6 (C-6); 127.7; 128.8 (C-2,6 Ph); 128.9 (C-3,5 Ph); 129.6 (C-4 Ph); 134.4; 141.5; 141.6; 150.7; 155.4; 159.3 (C-2); 160.3 (C-5). Found, %: C 50.96; H 4.18; N 12.99. $C_{19}H_{17}IN_4O$. Calculated, %: C 51.37; H 3.86; N 12.61.

(*S*)-1-(Adamantan-1-yl)-*N*-[(5-methyl-7-phenyloxazolo-[5,4-*b*]pyridin-2-yl)methyl]ethan-1-amine (12e). Yield 191 mg (51%), light-beige crystals, mp 157–160°C. IR spectrum, ν, cm^{-1}: 1619 (C=N), 2846, 2910 (C–H). ^{1}H NMR spectrum, δ, ppm: 1.00 (3H, d, J = 6.1, NHCHC<u>H</u>$_3$); 1.60–1.67 (12H, m, 6CH_2 Ad); 1.97 (3H, br. s, 3,5,7-CH Ad); 2.20 (1H, q, J = 6.1, 1-CH Ad); 2.68 (3H, s, CH_3); 4.05 (1H, d, J = 15.3) и 4.16 (1H, d, J = 15.2, NHC<u>H</u>$_2$); 5.29 (1H, br. s, NH); 7.36 (1H, s, H-6); 7.49–7.53 (3H, m, H-3,4,5 Ph); 8.06 (2H, d, J = 7.6, H-2,6 Ph). ^{13}C NMR

spectrum, δ, ppm.: 13.3 (2-CH_3); 24.3 (CH_3); 28.5 (3,5,7-CH Ad); 36.1 (1-C Ad); 37.2 (4,6,10-CH_2 Ad); 38.5 (2,8,9-CH_2 Ad); 45.7 ($NHCH_2$); 61.6 (CHN); 118.2 (C-6); 127.8; 128.8 (C-2,6 Ph); 128.9 (C-3,5 Ph); 129.4 (C-4 Ph); 134.8; 140.7; 154.2; 160.4 (C-2); 165.1 (C-5). Found, %: C 77.29; H 8.18; N 10.87. $C_{26}H_{31}N_3O$. Calculated, %: C 77.77; H 7.78; N 10.46.

6-Methyl-1-[(5-methyl-7-phenyloxazolo[5,4-*b*]pyridin-2-yl)methyl]-8-phenyl-1*H*-pyrido[2,3-*b*][1,4]oxazin-2(3*H*)-one (12f). Yield 185 mg (40%), beige crystals, mp 207–209°C. IR spectrum, ν, cm^{-1}: 1620 (C=N), 1699 (C=O), 2953, 2988 (C–H). ^{1}H NMR spectrum, δ, ppm: 2.46 (3H, s, CH_3); 2.63 (3H, s, CH_3); 4.74 (2H, s, NCH_2); 4.86 (2H, s, OCH_2); 6.77 (1H, s, H-7'); 7.28–7.31 (5H, m, H 8'-Ph); 7.33 (1H, s, H-6); 7.43 (1H, t, *J* = 7.6, H-4 7-Ph); 7.48 (2H, t, *J* = 7.6, H-3,5 7-Ph); 7.90 (2H, d, *J* =7.6, H-2,6 7-Ph). ^{13}C NMR spectrum, δ, ppm: 23.4 (CH_3); 24.3 (CH_3); 42.2 (NCH_2); 68.1 (OCH_2); 118.0 (H-6); 118.9 (C-8a'); 121.1 (C-7'); 127.5; 128.2; 128.6; 128.8; 129.0; 129.3 (C-4 7-Ph); 129.5 (C-4 8'-Ph); 134.2; 136.6; 140.4; 141.1; 152.4; 154.5; 155.2; 159.4; 160.0; 165.9 (C=O). Mass spectrum, *m*/*z* (I_{rel}, %): 462 $[M]^+$ (8), 200 (100), 199 (64), 171 (25), 100 (15). Found, *m*/*z*: 462.1686 $[M]^+$. $C_{28}H_{22}N_4O_3$. Calculated, *m*/*z*: 462.1688. Found, %: C 72.28; H 5.17; N 12.53. $C_{28}H_{22}N_4O_3$. Calculated, %: C 72.71; H 4.79; N 12.11.

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