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Dedicated to Full Member of the Russian Academy of Sciences G.A. Tolstikov on his 75th anniversary

One-Pot Synthesis of 6*H*-Pyrrolo[2,3-*e*][1,2,4]triazolo-[1,5-*a*]pyrimidines on the Basis of σ^H-Adducts of 6-Nitro[1,2,4]-triazolo[1,5-*a*]pyrimidine with Carbonyl Compounds

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Abstract—Aromatic, heteroaromatic, and aliphatic carbonyl compounds reacted with 6-nitro[1,2,4]triazolo-[1,5-*a*]pyrimidine in the presence of a base to give stable σ^H-adducts at C^7. Reduction of the nitro group in the latter is accompanied by tandem autoaromatization of the azine ring and intramolecular cyclocondensation with formation of the corresponding 6*H*-pyrrolo[2,3-*e*][1,2,4]triazolo[1,5-*a*]pyrimidines.

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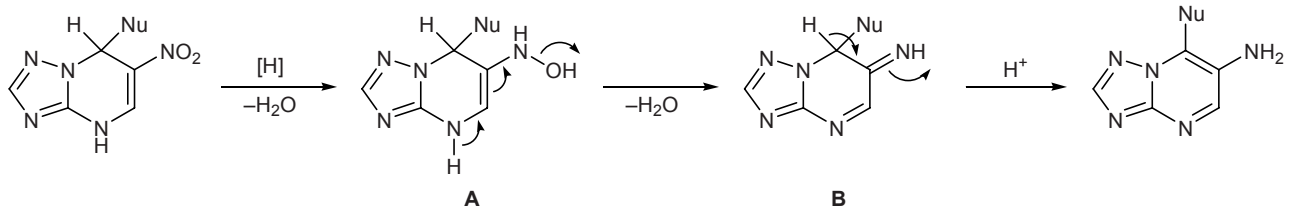
Important methods for building up fused pyrrole derivatives are based on the use of nitro-substituted aromatic and heteroaromatic compounds as starting materials. These procedures include introduction of an appropriate side chain into the *ortho* position with respect to the nitro group, reduction of the latter to amino, and cyclocondensation involving the amino group and the side chain. The most general examples of formation of pyrrole ring on the basis of nitroarenes (hetarenes) are intramolecular reactions of the amino group with electrophilic moiety of the side chain, e.g., carbonyl-containing group (Reissert reaction [1]) or acetonitrile, nitroethylene [2, 3], and vinyl or acetylene frgments [4].

Extensively developing approaches based on nucleophilic substitution of hydrogen (S_N^H reactions) make it possible to introduce into a nitroarene or hetarene molecule various fragments, including those capable of participating in subsequent cyclizations, via nucleophilic attack on unsubstituted carbon atom. Such reactions were successfully performed on a series of S_N^H products derived from nitroarenes with a view to synthesize indole derivatives [6, 7], as well as on 3-nitropyridines [8].

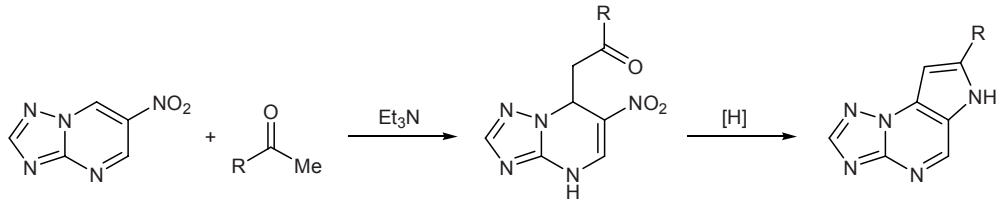
6-Nitroazolopyrimidines are capable of taking up under mild conditions aromatic (*N*,*N*-dialkylanilines, phenols, phenyl ethers) and heteroaromatic nucleophiles (indoles, pyrroles) and anions derived from some CH acids (methylazinium cations, malononitrile, ethyl cyanoacetate, etc. [9]). In most cases, these reactions stopped at the stage of formation of so stable σ^H-adducts that their subsequent oxidative aromatization was almost impossible.

We briefly reported previously that σ^H-adducts derived from 6-nitro[1,2,4]triazolo[1,5-*a*]pyrimidine (**I**) undergo unexpected autoaromatization of the pyrimi-

Scheme 1.



Scheme 2.



I **IIa–IIf** **IIIa–IIIf** **IVa–IVf**

R = 2-furyl (**a**), 2-thienyl (**b**), 2-(2-thienyl)ethenyl (**c**), PhCH=CH (**d**), 4-($ClCH_2CH_2$)C_6H_4 (**e**), 3,4-$CH_2O_2C_6H_4$ (**f**).

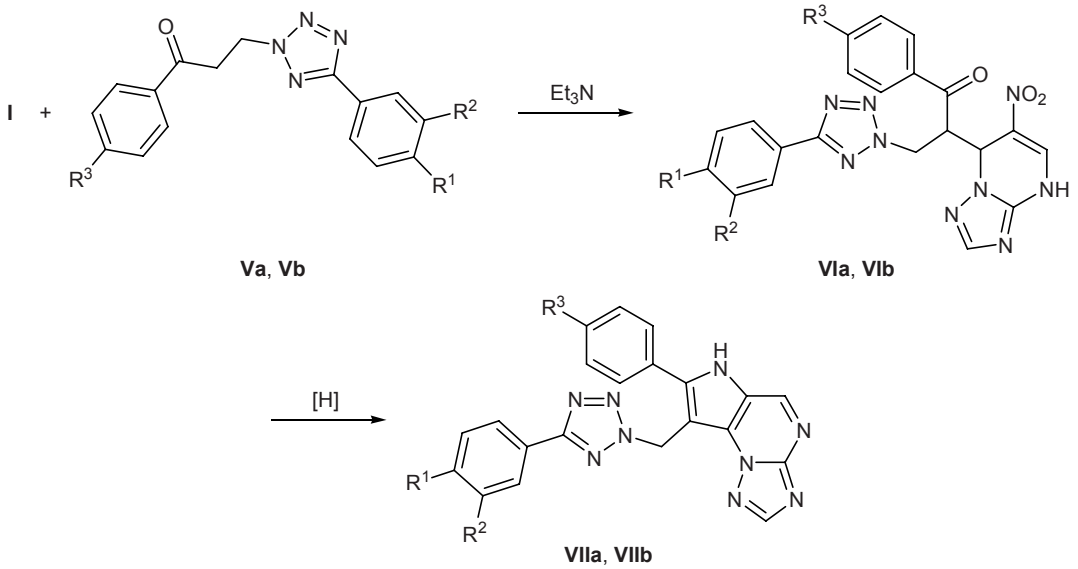
dine ring in the course of reduction of the nitro group [10]. It is known [5] that oxidative aromatization of σ^H-adducts formed from dihydroazines, by contrast, follows the $S_N^H(AO)$ mechanism. Alternatives are elimination mechanisms $S_N^H(AE)$ implying assistance by a readily departing group present in the substrate (*tele* or *cine* substitution), introduced into the σ^H-adduct with nucleophile (vicarious replacement), or formed during the process. In our case we tend to prefer the last version of *cine* substitution in hydroxylamine **A** which is formed as intermediate in the course of the reduction process (Scheme 1).

The presence in the resulting imine **B** molecule of two potential reaction centers in the *ortho* position with respect to each other creates favorable conditions for subsequent heterocyclization. In the present work we examined the possibility for synthesizing pyrrole-fused systems on the basis of σ^H-adducts derived from 6-nitro[1,2,4]triazolo[1,5-*a*]pyrimidine (**I**) via reaction with carbonyl compounds.

We found that 6-nitro[1,2,4]triazolo[1,5-*a*]pyrimidine (**I**) reacts with methyl ketones **IIa**–**IIf** to give stable adducts at C^7 (compounds **IIIa**–**IIIf**) in high yields (Scheme 2). The nitro group in σ^H-adducts **IIIa**–**IIIf** can be reduced by the action of various reducing agents, such as iron(II) hydroxide, tin(II) chloride, sodium dithionite, and hydrogen over Pd/C catalyst. The reduction is accompanied by aromatization of the pyrimidine ring and intramolecular ring closure to produce 6*H*-pyrrolo[2,3-*e*][1,2,4]triazolo[1,5-*a*]pyrimidines **IV** (Scheme 2). The best results were obtained using tin(II) chloride in the presence of concentrated hydrochloric acid.

The structure of σ^H-adducts **IIIa**–**IIIf** was determined on the basis of their ^1H NMR spectra and elemental analyses. Compounds **IIIa**–**IIIf** characteristically showed in the ^1H NMR spectra a signal from the 7-H proton as a triplet at δ 6.12–6.14 ppm, protons in the exocyclic CH_2 group resonated at δ 3.76–3.78 ppm as a doublet of doublets, and the 5-H and 2-H signals

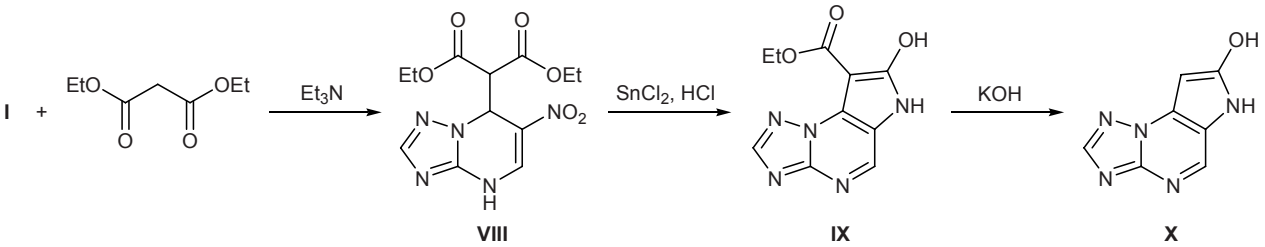
Scheme 3.



Va, **Vb** **VIa**, **VIb** **VIIa**, **VIIb**

$R^1 = R^2 = H$, $R^3 = MeO$ (**a**); $R^1 = R^2 = R^3 = MeO$ (**b**).

Scheme 4.



appeared as singlets at δ 8.37–8.39 and 7.77–7.78 ppm, respectively. In addition, signals from protons in the aryl substituent were present. The ^{1}H NMR spectra of tricyclic products **IVa**–**IVf** lacked 7-H signals, indicating that the pyrimidine ring therein is aromatic; the 8-H proton in the pyrrole ring gave a singlet at δ 7.4 ppm, singlets at δ 8.52–8.69 and 8.87–9.07 ppm were assigned to 2-H and 5-H, respectively, and signals from the substituent at C^7 were observed.

The proposed procedure is general; it ensures pyrrole annulation with both bulkier polyfunctional carbonyl compounds, e.g., ketones **V** having a pharmacophoric tetrazole fragment [11] (Scheme 3), and β-dicarbonyl compounds, in particular diethyl malonate (Scheme 4). Under the conditions analogous to the synthesis of adducts **IIIa**–**IIIf**, the reactions of compound **I** with ketones **Va** and **Vb** afforded stable σ^H-adducts **VIa** and **VIb** whose reduction with tin(II) chloride resulted in the formation of pyrrolotriazolopyrimidines **VIIa** and **VIIb**, respectively in 35–36% yield (Scheme 3). Compounds **VIa** and **VIb** displayed in the ^{1}H NMR spectra double sets of signals due to the presence of mixtures of diastereoisomers: the 7-H signals were observed as doublets at δ 6.44–6.33 ppm, the 5-H and 2-H protons gave rise to two singlets at δ 8.09–8.06 and 7.83–7.75, respectively, and the exocyclic CH and CH_2 protons resonated as multiplets at δ 5.27–5.16 and 5.00–4.68 ppm, respectively; in addition, signals from protons in the aromatic rings were present. The ^{1}H NMR spectra of reduction products **VIIa** and **VIIb** were considerably simpler, for their molecules lack chiral centers; the spectra contained a set of aromatic proton signals, singlets from 2-H and 5-H at δ 9.02–8.99 and 8.52–8.36 ppm, respectively, and a two-proton singlet at δ 6.3 ppm, corresponding to the CH_2 group.

The reduction of adduct **VIII** obtained from compound **I** and diethyl malonate gave ester **IX**, and alkaline hydrolysis of **IX**, followed by decarboxylation, lead to hydroxy derivative **X** (Scheme 4).

Thus the proposed procedure for the synthesis of pyrrolotriazolopyrimidines is attractive from the preparative viewpoint, for it ensures one-pot transformation of σ^H-adducts **III**, **VI**, and **VIII** into the target tricyclic structures, bypassing the oxidation step. Moreover, high electrophilicity of initial 6-nitro[1,2,4]triazolo[1,5-*a*]pyrimidine (**I**) ensures its successful reactions with various nucleophiles, which considerably extends the scope of application of the developed procedure.

EXPERIMENTAL

The progress of reactions and the purity of products were monitored by TLC on Armsorb KSGK-UF plates using chloroform–methanol (9:1) as eluent; detection under UV light. The ^{1}H NMR spectra were measured on a Bruker DRX-400 spectrometer (400 MHz) from solutions in DMSO-d_6 using tetramethylsilane as internal reference. The elemental compositions were determined on a Perkin–Elmer 240 automatic CHNO analyzer. The melting points were determined on a Boetius hot stage; uncorrected values are given.

7-Substituted 6-nitro-4,7-dihydro[1,2,4]triazolo-[1,5-*a*]pyrimidines IIIa–IIIf, VIa, VIb, and VIII (*general procedure*). A mixture of 1 mmol of triazolopyrimidine **I**, 1 mmol of carbonyl compound **Ia**–**If**, **Va**, or **Vb** or diethyl malonate, and 140 μl (1 mmol) of triethylamine in 3 ml of acetonitrile was kept for 24 h at room temperature. The mixture was acidified with concentrated hydrochloric acid, and the precipitate was filtered off, washed with distilled water and acetonitrile, and dried in air.

1-(2-Furyl)-2-(6-nitro-4,7-dihydro[1,2,4]triazolo-[1,5-*a*]pyrimidin-7-yl)ethanone (IIIa). Yield 76%, mp 236–238°C. ^{1}H NMR spectrum, δ, ppm: 11.80 s (NH), 8.26 s (5-H), 7.82 d.d (1H, H_{arom}), 7.80 s (2-H), 7.27 d.d (1H, H_{arom}), 6.58 d.d (1H, H_{arom}), 6.05 t (7-H), 3.55 d (2H, CH_2). Found, %: C 47.90; H 3.48; N 25.40. $C_{11}H_9N_5O_4$. Calculated, %: C 48.01; H 3.30; N 25.45.

2-(6-Nitro-4,7-dihydro[1,2,4]triazolo[1,5-*a*]pyrimidin-7-yl)-1-(2-thienyl)ethanone (IIIb). Yield 71%,

mp 200–204°C. ^{1}H NMR spectrum, δ, ppm: 11.83 s (NH), 8.25 s (5-H), 7.85 d.d (1H, H_{arom}), 7.79 s (2-H), 7.77 d.d (1H, H_{arom}), 7.14 d.d (1H, H_{arom}), 5.99 t (7-H), 3.66 d (2H, CH_2). Found, %: C 45.28; H 3.12; N 24.10. $C_{11}H_9N_5O_3S$. Calculated, %: C 45.36; H 3.11; N 24.04.

1-(6-Nitro-4,7-dihydro[1,2,4]triazolo[1,5-*a*]pyrimidin-7-yl)-4-(2-thienyl)but-3-en-2-one (IIIc). Yield 76%, mp 250–252°C. ^{1}H NMR spectrum, δ, ppm: 11.81 s (NH), 8.26 s (5-H), 7.68 d.d (1H, H_{arom}), 7.64 d (1H, CH), 7.61 d.d (1H, H_{arom}), 7.43 s (2-H), 7.09 d.d (1H, H_{arom}), 6.33 d (1H, CH), 5.98 t (7-H), 3.43 d (2H, CH_2). Found, %: C 49.22; H 3.44; N 22.15. $C_{13}H_{11}N_5O_3S$. Calculated, %: C 49.21; H 3.49; N 22.07.

1-(6-Nitro-4,7-dihydro[1,2,4]triazolo[1,5-*a*]pyrimidin-7-yl)-4-phenylbut-3-en-2-one (IIId). Yield 69%, mp 270–272°C. ^{1}H NMR spectrum, δ, ppm: 12.19 s (NH), 8.25 s (5-H), 7.67 s (2-H), 7.58 d (1H, CH), 7.50–7.47 m (1H, H_{arom}), 7.34–7.30 m (2H, H_{arom}), 6.97–6.95 m (2H, H_{arom}), 6.49 d (1H, CH), 5.99 t (7-H), 3.45 d (2H, CH_2). Found, %: C 57.73; H 4.22; N 22.55. $C_{15}H_{13}N_5O_3$. Calculated, %: C 57.88; H 4.21; N 22.50.

1-[4-(2-Chloroethyl)phenyl]-2-(6-nitro-4,7-dihydro[1,2,4]triazolo[1,5-*a*]pyrimidin-7-yl)ethanone (IIIe). Yield 77%, mp 230–234°C. ^{1}H NMR spectrum, δ, ppm: 11.78 s (NH), 8.23 s (5-H), 7.67 s (2-H), 7.87 d (2H, H_{arom}), 7.66 d (2H, H_{arom}), 5.91 t (7-H), 4.33 t (2H, CH_2), 3.38 d (2H, CH_2), 3.34 t (2H, CH_2). Found, %: C 51.71; H 4.06; N 20.15. $C_{15}H_{14}ClN_5O_3$. Calculated, %: C 51.81; H 4.06; N 20.14.

1-(1,3-Benzodioxol-5-yl)-2-(6-nitro-4,7-dihydro[1,2,4]triazolo[1,5-*a*]pyrimidin-7-yl)ethanone (IIIf). Yield 51%, mp 238–240°C. ^{1}H NMR spectrum, δ, ppm: 11.89 s (NH), 8.37 s (5-H), 7.77 s (2-H), 7.82 d (1H, H_{arom}), 7.67–7.65 m (2H, H_{arom}), 6.12 t (7-H), 4.33 t (2H, CH_2), 5.98 s (2H, CH_2). Found, %: C 51.00; H 3.40; N 21.23. $C_{14}H_{11}N_5O_5$. Calculated, %: C 51.07; H 3.37; N 21.23.

1-(3-Methoxyphenyl)-2-(6-nitro-4,7-dihydro[1,2,4]triazolo[1,5-*a*]pyrimidin-7-yl)-3-(5-phenyl-2*H*-tetrazol-2-yl)propan-1-one (VIa). Yield 78%, mp 234–235°C. ^{1}H NMR spectrum, δ, ppm: 12.00 s (NH), 8.09 s (1H, 5-H), 8.02 s (0.75H, 5-H), 7.99–7.97 m (3H, H_{arom}, 2-H), 7.92 d (1.55H, H_{arom}), 7.83 s (0.72H, 2-H), 7.74 d (2H, H_{arom}), 7.49–7.45 m (5H, H_{arom}), 7.02 d (2H, H_{arom}), 6.96 d (1.51H, H_{arom}), 6.44 d (1H, 7-H), 6.33 d (0.73H, 7-H), 5.21–5.18 m (1H, CH), 5.03–4.98 m (1H, CH_2), 4.76–4.75 (1H, CH_2), 3.87 s (3H, CH_3), 3.84 s (2.24H, CH_3). Found, %: C 55.90; H 4.05; N 26.78. $C_{22}H_{19}N_9O_4$. Calculated, %: C 55.81; H 4.05; N 26.63.

3-[5-(3,4-Dimethoxyphenyl)-2*H*-tetrazol-2-yl]-1-(3-methoxyphenyl)-2-(6-nitro-4,7-dihydro[1,2,4]-triazolo[1,5-*a*]pyrimidin-7-yl)propan-1-one (VIb). Yield 85%, mp 152–154°C. ^{1}H NMR spectrum, δ, ppm: 11.95 s (NH), 8.06 s (1H, 5-H), 8.02 s (0.23H, 5-H), 7.98 d (2H, H_{arom}), 7.75 t (1H, H_{arom}), 7.53 d.d (1H, H_{arom}), 7.43 s (1H, 2-H), 7.40 s (0.26H, 2-H), 7.03 d (2H, H_{arom}), 6.99–6.95 m (1H, H_{arom}), 6.43 d (1H, 7-H), 6.33 d (0.21H, 7-H), 5.18–5.17 m (1H, CH), 5.02–4.97 m (1H, CH_2), 4.73–4.68 (1H, CH_2), 3.87 s (3H, CH_3), 3.84 s (6H, CH_3), 3.82 s (0.8H, CH_3), 3.79 s (0.7H, CH_3). Found, %: C 53.97; H 4.40; N 23.60. $C_{24}H_{23}N_9O_6$. Calculated, %: C 54.03; H 4.35; N 23.63.

Diethyl 2-(6-nitro-4,7-dihydro[1,2,4]triazolo-[1,5-*a*]pyrimidin-7-yl)malonate (VIII). Yield 78%, mp 145–147°C. Found, %: C 44.30; H 4.63; N 21.54. $C_{12}H_{15}N_5O_6$. Calculated, %: C 44.31; H 4.65; N 21.53.

7-Substituted pyrrolo[2,3-*e*][1,2,4]triazolo[1,5-*a*]-pyrimidines IVa–IVf, VIIa, VIIb, and IX (*general procedure***).** A solution of 290 mg of tin(II) chloride dihydrate in 2 ml of acetic acid and 250 μl of concentrated hydrochloric acid were added under stirring to a solution of 100 mg of compound **IIIa**–**IIIf**, **VIa**, **VIb**, or **VIII** in 3 ml of acetic acid. The precipitate was filtered off, washed with distilled water, and dissolved in DMF, the solution was applied to aluminum oxide, the product was washed off with chloroform–ethanol (3 : 1), and the filtrate was evaporated.

7-(2-Furyl)-6*H*-pyrrolo[2,3-*e*][1,2,4]triazolo-[1,5-*a*]pyrimidine (IVa). Yield 34%, mp > 300°C. ^{1}H NMR spectrum, δ, ppm: 13.13 s (NH), 8.95 s (5-H), 8.54 s (2-H), 7.85 s (8-H), 7.26–7.22 m (2H, H_{arom}), 7.61–4.76 m (1H, H_{arom}). Found, %: C 58.59; H 3.10; N 31.02. $C_{11}H_7N_5O$. Calculated, %: C 58.67; H 3.13; N 31.02.

7-(2-Thienyl)-6*H*-pyrrolo[2,3-*e*][1,2,4]triazolo-[1,5-*a*]pyrimidine (IVb). Yield 37%, mp > 300°C. ^{1}H NMR spectrum, δ, ppm: 13.18 s (NH), 8.94 s (5-H), 8.52 s (2-H), 7.62 d.d (1H, H_{arom}), 7.30 s (8-H), 7.18–7.12 m (2H, H_{arom}). Found, %: C 54.70; H 3.00; N 29.00. $C_{11}H_7N_5S$. Calculated, %: C 54.76; H 2.92; N 29.03.

7-[2-(2-Thienyl)vinyl]-6*H*-pyrrolo[2,3-*e*][1,2,4]-triazolo[1,5-*a*]pyrimidine (IVc). Yield 32%, mp > 300°C. ^{1}H NMR spectrum, δ, ppm: 12.74 s (NH), 8.94 s (5-H), 8.50 s (2-H), 8.78 d (1H, CH), 7.62 d.d (1H, H_{arom}), 7.34 s (8-H), 7.16–7.14 m (2H, H_{arom}),

7.07 d (1H, CH). Found, %: C 58.36; H 3.34; N 26.11. $C_{13}H_9N_5S$. Calculated, %: C 58.41; H 3.39; N 26.20.

7-(2-Phenylethenyl)-6*H*-pyrrolo[2,3-*e*][1,2,4]-triazolo[1,5-*a*]pyrimidine (IVd). Yield 38%, mp > 300°C. ^{1}H NMR spectrum, δ, ppm: 13.18 s (NH), 8.93 s (5-H), 7.49 s (2-H), 7.82 d (2H, H_{arom}), 7.76 d (2H, H_{arom}), 7.10 s (8-H), 3.38 d (2H, CH_2), 3.34 t (2H, CH_2). Found, %: C 69.03; H 4.21; N 26.83. $C_{15}H_{11}N_5$. Calculated, %: C 68.95; H 4.24; N 26.83.

7-[4-(2-Chloroethyl)phenyl]-6*H*-pyrrolo[2,3-*e*]-[1,2,4]triazolo[1,5-*a*]pyrimidine (IVe). Yield 34%, mp > 300°C. ^{1}H NMR spectrum, δ, ppm: 13.04 s (NH), 9.00 s (5-H), 8.68 s (2-H), 7.58–7.56 m (2H, H_{arom}), 7.17 s (8-H), 7.39–7.37 m (3H, H_{arom}), 7.49 d (1H, CH), 6.65 d (1H, CH). Found, %: C 60.46; H 4.00; N 23.48. $C_{15}H_{12}ClN_5$. Calculated, %: C 60.51; H 4.00; N 23.48

7-(1,3-Benzodioxol-5-yl)-6*H*-pyrrolo[2,3-*e*]-[1,2,4]triazolo[1,5-*a*]pyrimidine (IVf). Yield 34%, mp > 300°C. ^{1}H NMR spectrum, δ, ppm: 13.03 s (NH), 8.82 s (5-H), 7.79 d (1H, H_{arom}), 7.70 s (2-H), 7.62–7.59 m (2H, H_{arom}), 5.98 s (2H, CH_2). Found, %: C 60.15; H 3.30; N 25.10. $C_{14}H_9N_5O_2$. Calculated, %: C 60.21; H 3.25; N 25.10.

7-(4-Methoxyphenyl)-8-(5-phenyl-2*H*-tetrazol-2-ylmethyl)-6*H*-pyrrolo[2,3-*e*][1,2,4]triazolo[1,5-*a*]-pyrimidine (VIIa). Yield 36%, mp 264–266°C. ^{1}H NMR spectrum, δ, ppm: 13.03 s (NH), 8.82 s (5-H), 7.79 d (1H, H_{arom}), 7.70 s (2-H), 7.62–7.59 m (2H, H_{arom}), 5.98 s (2H, CH_2). Found, %: C 62.36; H 4.00; N 25.79. $C_{22}H_{17}N_9O$. Calculated, %: C 62.40; H 4.05; N 29.79.

8-[5-(3,4-Dimethoxyphenyl)-2*H*-tetrazol-2-ylmethyl]-7-(4-methoxyphenyl)-6*H*-pyrrolo[2,3-*e*]-[1,2,4]triazolo[1,5-*a*]pyrimidine (VIIb). Yield 40%, mp > 300°C. ^{1}H NMR spectrum, δ, ppm: 13.00 s (NH), 9.03 s (5-H), 8.52 s (2-H), 7.75 d (2H, H_{arom}), 7.51 d.d (1H, H_{arom}), 7.44 d (1H, H_{arom}), 7.16 d (2H, H_{arom}), 7.07 d (1H, H_{arom}), 6.39 s (2H, CH_2), 3.85 s (3H, CH_3), 3.80 s (3H, CH_3), 3.79 s (3H, CH_3). Found, %: C 48.50; H 3.59; N 28.34. $C_{24}H_{21}N_9O_3$. Calculated, %: C 48.59; H 3.67; N 28.33.

Ethyl 7-hydroxy-6*H*-pyrrolo[2,3-*e*][1,2,4]triazolo[1,5-*a*]pyrimidine-8-carboxylate (IX). Yield 40%, mp > 300°C. ^{1}H NMR spectrum, δ, ppm: 13.00 s (NH), 9.03 s (5-H), 8.32 s (2-H), 6.22 d (7-H), 4.23–4.12 q (2H, CH_2), 4.07–4.00 q (2H, CH_2), 1.30 t (3H, CH_3), 1.16 t (3H, CH_3). Found, %: C 48.50; H 3.59; N 28.34. $C_{24}H_{21}N_9O_3$. Calculated, %: C 48.59; H 3.67; N 28.33.

7-Hydroxy[1,2,4]triazolo[1,5-*a*]pyrrolo[2,3-*e*]-pyrimidine (X). A mixture of 1 mmol of ester **IX** and a 2 M solution of potassium hydroxide was heated for 30 min at the boiling point. The mixture was cooled and neutralized with acetic acid, and the precipitate was filtered off, repeatedly washed with water, and dried in air. Yield 67%, mp > 300°C. ^{1}H NMR spectrum, δ, ppm: 12.74 s (NH), 10.27 s (OH), 8.27 s (5-H), 7.31 s (2-H), 5.55 s (8-H). Found, %: C 48.09; H 2.99; N 39.97. $C_7H_5N_5O$. Calculated, %: C 48.00; H 2.88; N 39.98.

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