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Alternative Routes to the Pyrazolo[4,3-*e*][1,2,4]triazolo-[1,5-*c*]pyrimidine System

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Abstract—Formimidic acid esters derived from 1-substituted 5-aminopyrazole-4-carbonitriles reacted with carboxylic acid hydrazides on heating to give 2,7-disubstituted pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine derivatives as a result of double heterocyclization. The same compounds were obtained by thermal recyclization of 1-aroyl-2-(1-R-pyrazolo[3,4-*d*]pyrimidin-4-yl)hydrazines.

It is known that treatment of 5-aminopyrazole-4-carbonitriles **I** with ethyl orthoformate gives formimidic acid esters **II** whose reactions with primary amines are accompanied by heterocyclization to afford pyrazolo[3,4-*d*]pyrimidine derivatives [1, 2]. The products attract interest from the viewpoint of their subsequent transformations [1–6] and exhibit a broad spectrum of biological activity [7–11]. We anticipated [12] that the use of carboxylic acid hydrazides as the amine component will give rise to cascade heterocyclization **II** → **III** → **IV** → **V** with formation of a new heterocyclic system, pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine derivatives **V**. In fact, formimidic acid esters **II** reacted with hydrazides under fairly severe conditions (prolonged heating in boiling bromobenzene) to give the expected compounds **V** in about 50% yield (isolated pure products). The proposed transformation sequence (Scheme 1) was confirmed experimentally using the reaction of ester **IIa** with *p*-methoxybenzohydrazide as an example. The reaction was carried out under milder conditions, and we succeeded in isolating intermediate compounds **IIIa** and **IVa** and hence converting in a stepwise mode amidrazone **IIIa** into pyrazolopyrimidine **IVa**, and the latter, into the target product **Va**.

Amidrazone **IIIa** was readily obtained by heating imidic ester **IIa** with *p*-methoxybenzohydrazide in alcohol. The IR spectrum of **IIIa** contained absorption bands due to stretching vibrations of the cyano (2200 cm^{-1}), carbonyl (1675 cm^{-1}), amidine (1650 cm^{-1}), and imino groups (3060, 3330, 3355 cm^{-1}). Compound **IIIa** showed in the mass spectrum the molecular ion peak $M_1^{+\cdot}$ (*m*/*z* 284) which underwent cascade heterocyclization with elimination of water molecule, resulting in formation of ion $M_2^{+\cdot}$ (*m*/*z* 266) corresponding to the molecular ion of pyrazolotriazolopyrimidine **Va**.

Heating of amidrazone **IIIa** in dimethylformamide for a short time led to closure of pyrimidine ring to afford 4-iminopyrazolopyrimidine **IVa**. The IR spectrum of **IVa** lacked C≡N absorption, the carbonyl stretching vibration frequency decreased to 1650 cm^{-1}, and the NH absorption pattern differed from that observed for **IIIa** (3115, 3200 cm^{-1}). Compounds **IIIa** and **IVa** are very poorly soluble in organic solvents, and we failed to record their ^{1}H NMR spectra. Nevertheless, the above IR data are consistent with the assumed structures.

Dehydration of iminopyrimidine **IVa** with closure of triazole ring (**IVa** → **Va**) was effected by prolonged heating in boiling bromobenzene. In the IR spectrum of compound **Va** dispersed in mineral oil we observed a fairly strong absorption band at 1650 cm^{-1}, whose origin is difficult to interpret. The structure of **Va** was confirmed by other spectral methods. Its ^{1}H NMR spectrum contained a three-proton singlet due to the methoxy group (δ 3.85 ppm), two two-proton doublet from the aromatic protons of the 2-*p*-methoxyphenyl substituent (δ 7.1 and 8.2 ppm), a sharp singlet at δ 9.5 ppm due to 5-H (C^2 in the pyrimidine ring), and two broadened signals from protons in the pyrazole ring (δ 8.6 and 14.4 ppm); broadening of the latter signals is likely to result from prototropic transformations in the pyrazole ring. The most abundant peak in the mass spectrum of **Va** was that of the molecular ion

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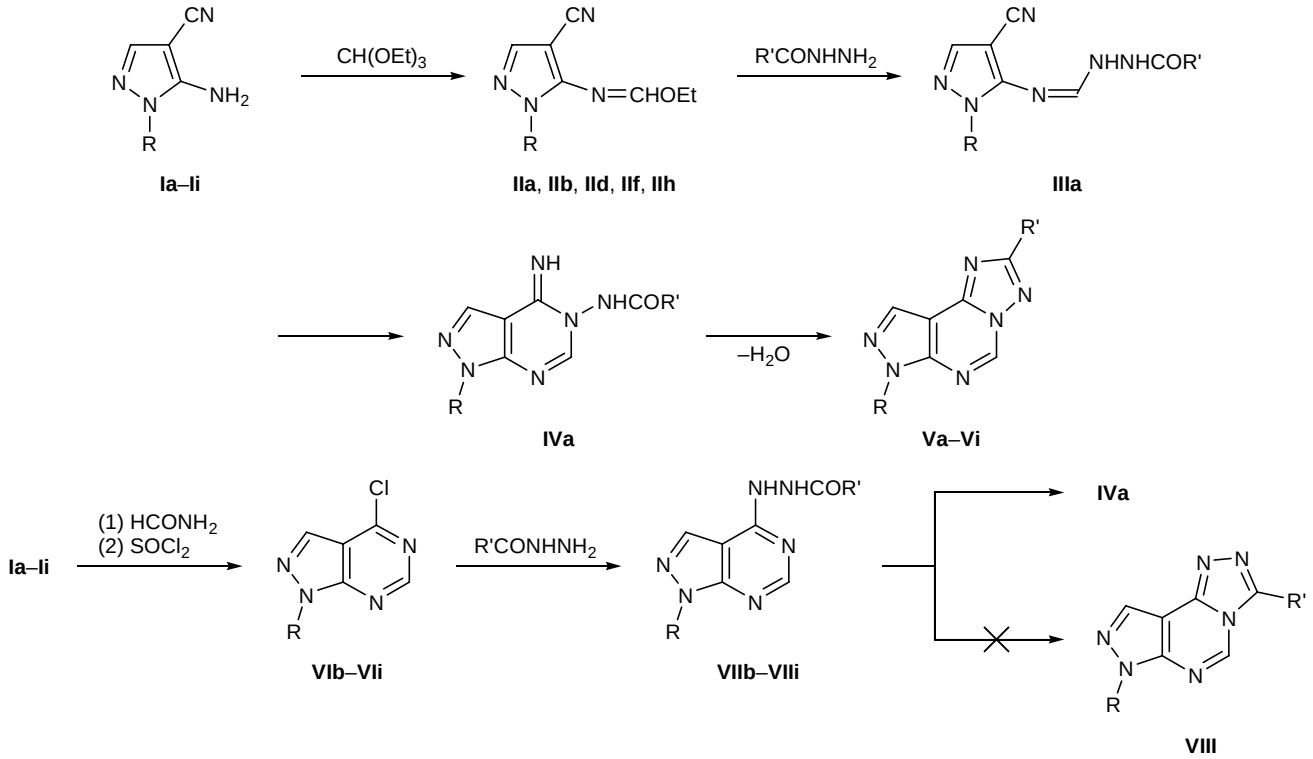
Scheme 1.



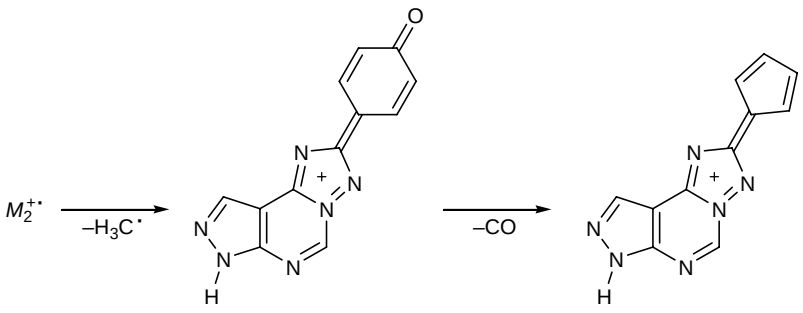
R = H, R' = 4-$MeOC_6H_4$ (**a**); R = Me, R' = Ph (**b**), 4-$MeOC_6H_4$ (**c**); R = $PhCH_2$, R' = Ph (**d**), 4-$MeOC_6H_4$ (**e**); R = Ph, R' = 4-$MeOC_6H_4$ (**f**), 4-$ClC_6H_4OCH_2$ (**g**); R = 4-MeC_6H_4, R' = Ph (**h**), 4-MeC_6H_4 (**i**).

$M_2^{+\cdot}$ with *m*/*z* 266; its subsequent fragmentation gives ions $\mathbf{F}_1$ (*m*/*z* 251) and $\mathbf{F}_2$ (*m*/*z* 223) via successive elimination of $H_3C^{\cdot}$ radical and CO molecule (Scheme 2). The spectral parameters of the other pyrazolo[4,3-*e*]-[1,2,4]triazolo[1,5-*c*]pyrimidines **Vb**–**Vi** did not contradict the assumed structure.

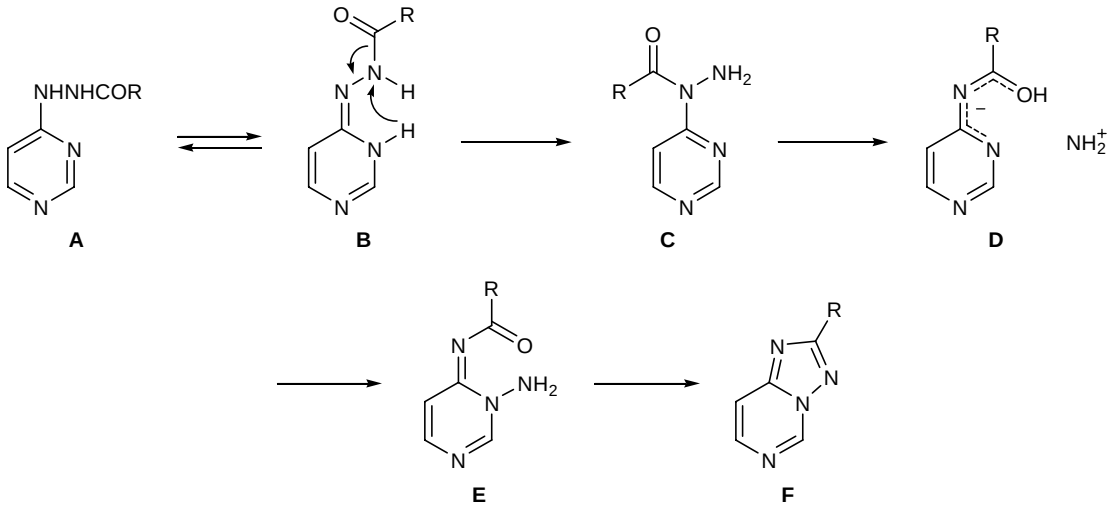
We also made an attempt to synthesize pyrazolo-[4,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidines **VIII** which are isomeric to **V**; they differ from **V** by the mode of junction of the triazole and pyrimidine rings. For this purpose, chloro derivatives **VI** prepared by the known scheme [3, 4] were brought into reaction with carboxylic acid hydrazides. As a result, we isolated previously unknown 4-acylhydrazinopyrazolo[3,4-*d*]pyrimidines **VII**.

According to the ^{1}H NMR data, compounds **VII** in DMSO-d_6 at 20°C exist in the amino rather than imino form: the spectra contained two broadened one-proton doublets in the region δ 9.5–11.0 ppm. In the spectrum recorded at elevated temperature, considerable broadening of signals from protons of the 4-hydrazino substituent was observed, and the patterns in the regions corresponding to aromatic protons and functional groups became simpler due to rupture of intramolec-

Scheme 2.



Scheme 3.



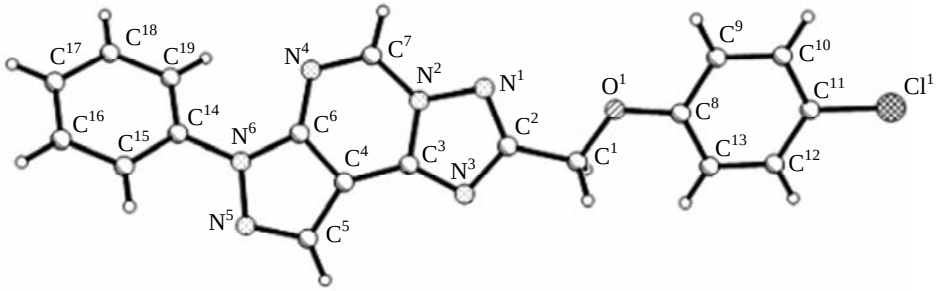
ular hydrogen bonds and acceleration of tautomeric and exchange processes.

Dehydration of hydrazides **VII** occurred under very severe conditions, on heating above their melting points. The ^{1}H NMR spectra of the dehydration products were fully identical to those of compounds **V**. Structure **V** (rather than **VIII**) was proved by the X-ray diffraction data for the product obtained from hydrazide **VIIg** (see figure). The geometric parameters of molecule **Vg** indicate essential conjugation in the tricyclic system. However, all formally double bonds (C^2–N^2 1.318, C^3–N^3 1.322, C^5–N^5 1.324, C^7–N^4 1.301 Å) are slightly shorter than formally single bonds (C^2–N^3 1.360, C^3–N^2 1.375, C^6–N^4 1.362, C^6–N^6 1.361, C^7–N^2 1.375 Å). The tricyclic system is planar, the C^{14}–C^{19} benzene ring is turned through a dihedral angle of 34.86(4)°, the O^1 atom deviates from the plane by 0.40 Å, and the C^8–C^{13} is turned through an angle of 20.57(5)° with respect to the plane of the fused heterocyclic skeleton.

Thus the observed heterocyclization of hydrazides **VII** is accompanied by rearrangement which can be regarded as the reverse to the known base-catalyzed Dimroth rearrangement [1]. Presumably, the rearrangement **VII** → **V** includes consecutive migration of proton and the acyl group (**B** → **C**) in the amidrazone fragment. Cleavage of the N–N bond in 1-acyl-1-hetarylhydrazine **C** gives intimate ion pair **D** consisting of resonance-stabilized anion and amino cation, which is then converted into *N*-amino derivative **E**. The mechanism of the latter stage is analogous to N-amination of nitrogen-containing heterocycles with hydroxylamine–sulfuric acid [13]. Dehydration of intermediate **E** gives final product **F** (Scheme 2).

EXPERIMENTAL

The IR spectra were recorded on a Specord 71IR spectrometer from samples dispersed in mineral oil. The ^{1}H NMR spectra were measured on a Unity-300 instrument (300 MHz) from solutions in DMSO-d_6 using HMDS as internal reference. The mass spectra (electron impact, 70 eV) were obtained on a Kratos mass spectrometer with direct sample admission into the ion source (control voltage 1.75 kV).



Structure of the molecule of 2-(4-chlorophenoxy)-7-phenyl-7*H*-pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine (**Vg**) according to the X-ray diffraction data.

X-Ray analysis of compound **Vg** was performed on a Bruker SMART CCD area detector at 120 K (λMoK_α irradiation, $2\theta_{max}$ = 58.00°). Total of 12416 reflections were measured from a 0.50×0.40×0.07-mm single crystal (colorless plates); $C_{19}H_{13}ClN_6O$ (*M* 376.80). Monoclinic crystals with the following unit cell parameters (120 K): *a* = 19.047(7), *b* = 12.206(5), *c* = 7.226(3) Å; β = 93.292(8)°; *V* = 1677(1) Å^3; space group $P2_1/c$; *Z* = 4, d_{calc} = 1.492 g/cm^3. Averaging of equivalent reflections gave 4436 independent reflections (R_{int} = 0.0531) which were used in the structure solution and refinement. Absorption (μ = 0.252 mm^{-1}) was not taken into account, and the parameters T_{max} and T_{min} (0.862 and 0.123, respectively) were calculated using SADABS program. The structure was solved by the direct method. All non-hydrogen atoms were localized from the difference synthesis of electron density, and their positions were refined with respect to F^2_{hkl} in anisotropic approximation. All hydrogen atoms were localized from the geometry considerations and were taken into account in the refinement using the rider model with *U*(H) = 1.2*U*(C), where *U*(C) is the equivalent temperature factor of the carbon atom to which the corresponding hydrogen atom is attached. The final divergence factors were R_1 = 0.0576 [calculated with respect to F_{hkl} from 3549 reflections with $I > 2\sigma(I)$] and wR_2 = 0.1693 (calculated with respect to F^2_{hkl} from all 4436 reflections); GOOF = 1.046; 244 refined parameters. All calculations were performed using SHELXTL PLUS 5 software package [14].

Initial 5-aminopyrazole-4-carbonitriles **I** were synthesized by reaction of ethoxymethylenemalonodinitrile with hydrazine hydrate or the corresponding monosubstituted hydrazines according to the procedure reported in [4]. Compounds **I** were converted into formimidic acid esters **II** by the action of triethyl orthoformate as described in [2]. Chloropyrazolopyrimidines **VI** were prepared by the procedure described in [3, 4].

***N′*-(4-Cyano-1*H*-pyrazol-5-yliminomethyl)-4-methoxybenzohydrazide (IIIa).** A solution of 83 mg (0.5 mmol) of *p*-methoxybenzohydrazide in 1 ml of ethanol was added to a solution of 82 mg (0.5 mmol) of ester **IIa** in 2 ml of ethanol. The mixture was kept for 1 h at room temperature, and the precipitate was filtered off and washed with alcohol and water. Yield 60 mg (0.21 mmol, 42.3%), mp 304–306°C. IR spectrum, ν, cm^{-1}: 3555, 3330, 3060, 2200, 1673, 1650. Found, %: C 54.71; H 4.35; N 29.63. *m*/*z* 284 $[M]^+$. $C_{13}H_{12}N_6O_2$. Calculated, %: C 54.92; H 4.23; N 29.58. *M* 284.

***N*-(4-Imino-4,5-dihydro-1*H*-pyrazolo[3,4-*d*]pyrimidin-5-yl)-*p*-methoxybenzamide (IVa).** A suspension of 60 mg (0.21 mmol) of compound **IIIa** in 2 ml of DMF was heated to the boiling point. The mixture became homogeneous, and, after 10 min, a solid precipitated. The product was filtered off and washed with hexane. Yield 50 mg (0.17 mmol, 92%), mp 345°C. IR spectrum, ν, cm^{-1}: 3200, 3115, 1650. Found, %: C 55.12; H 4.35; N 29.71. $C_{13}H_{12}N_6O_2$. Calculated, %: C 54.92; H 4.23; N 29.58.

2-(4-Methoxyphenyl)-7*H*-pyrazolo[4,3-*e*]triazolo[1,5-*c*]pyrimidine (Va). A suspension of 100 mg (0.35 mol) of compound **IVa** in 2 ml of bromobenzene was heated for 10 h under reflux. The mixture was cooled, and the colorless precipitate was filtered off. Yield 60 mg (0.225 mmol, 64%), mp 274–275°C. IR spectrum, ν, cm^{-1}: 3167, 3100, 1650. ^{1}H NMR spectrum, δ, ppm: 3.85 s (3H), 7.1 d (2H), 8.2 d (2H), 8.6 br.s (1H), 14.4 br.s (1H). Found, %: C 58.78; H 3.83; N 31.46. *m*/*z* 266 $[M]^+$. $C_{13}H_{10}N_6O$. Calculated, %: C 58.65; H 3.76; N 31.58.

2,7-Disubstituted 7*H*-pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidines Vb–Vi. *a.* A mixture of equimolar amounts of compound **IIb**, **IId**, or **IIh** and the corresponding carboxylic acid hydrazide in bromobenzene was heated for 5–10 h at the boiling point. The mixture was cooled, and the precipitate was filtered off and recrystallized from DMF.

b. Pyrazolo[3,4-*d*]pyrimidine **VIIb–VIIi** was heated at the meting point until water vapor no longer evolved. After cooling, the product solidified and was recrystallized from DMF.

7-Methyl-2-phenyl-7*H*-pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine (Vb). Yield 45 (*a*), 75% (*b*), mp 245–247°C. ^{1}H NMR spectrum, δ, ppm: 4.2 s (3H), 7.5–8.3 m (5H), 8.4 s (1H), 9.1 s (1H). Found, %: C 62.3; H 4.0; N 33.75. $C_{13}H_{10}N_6$. Calculated, %: C 62.4; H 4.0; N 33.6.

2-(4-Methoxyphenyl)-7-methyl-7*H*-pyrazolo-[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine (Vc). Yield 39 (*a*), 65% (*b*), mp 241–242°C. ^{1}H NMR spectrum, δ, ppm: 3.9 s (3H), 4.2 s (3H), 7.0–8.0 m (4H), 8.4 s (1H), 9.1 s (1H). Found, %: C 60.3; H 4.1; N 29.9. $C_{14}H_{12}N_6O$. Calculated, %: C 60.04; H 4.3; N 30.0.

7-Benzyl-2-phenyl-7*H*-pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine (Vd). Yield 42 (*a*), 65% (*b*), mp 276–278°C. ^{1}H NMR spectrum, δ, ppm: 5.7 s (2H), 7.3–8.3 m (10H), 8.4 s (1H), 9.1 s (1H). Found, %: C 69.5; H 4.15; N 25.8. $C_{19}H_{14}N_6$. Calculated, %: C 69.944; H 4.3; N 25.77.

7-Benzyl-(4-methoxyphenyl)-7*H*-pyrazolo[4,3-*e*]-[1,2,4]triazolo[1,5-*c*]pyrimidine (Ve). Yield 54 (*a*), 76% (*b*), mp 207–208°C. ^{1}H NMR spectrum, δ, ppm: 3.9 s (3H), 5.7 s (2H), 7.0–7.4 m (9H), 8.4 s (1H), 9.2 s (1H). Found, %: C 67.5; H 4.2; N 23.59. $C_{20}H_{16}N_6O$. Calculated, %: C 67.42; H 4.49; N 23.6.

2-(4-Methoxyphenyl)-7-phenyl-7*H*-pyrazolo-[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine (Vf). Yield 43 (*a*), 72% (b), mp 232–233°C. ^{1}H NMR spectrum, δ, ppm: 7.4–8.4 m (12H), 8.6 s (1H), 9.2 s (1H). Found, %: C 66.5; H 4.1; N 24.6. $C_{19}H_{14}N_6O$. Calculated, %: C 66.66; H 4.03; N 24.56.0.

2-(4-Chlorophenoxy)-7-phenyl-7*H*-pyrazolo-[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine (Vg). Yield 58 (*a*), 70% (*b*), mp 188–190°C. ^{1}H NMR spectrum, δ, ppm: 5.4 s (2H), 7.0–8.2 m (9H), 8.5 s (1H), 9.2 s (1H). Found, %: C 60.72; H 3.56; Cl 9.62; N 22.46. $C_{19}H_{13}ClN_6O$. Calculated, %: C 60.56; H 3.45; Cl 9.43; N 22.31.

2-Phenyl-7-*p*-tolyl-7*H*-pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine (Vh). Yield 38 (*a*), 66% (*b*), mp 282°C. ^{1}H NMR spectrum, δ, ppm: 2.4 s (3H), 7.4–8.4 m (9H), 8.6 s (1H), 9.2 s (1H). Found, %: C 70.15; H 4.16; N 25.91. $C_{19}H_{14}N_6$. Calculated, %: C 69.94; H 4.29; N 25.77.

2,7-Di-*p*-tolyl-7*H*-pyrazolo[4,3-*e*][1,2,4]triazolo-[1,5-*c*]pyrimidine (Vi). Yield 56 (*a*), 71% (*b*), mp 310°C. ^{1}H NMR spectrum, δ, ppm: 2.4 s (6H), 7.3–8.2 m (8H), 8.6 s (1H), 9.2 s (1H). Found, %: C 70.72; H 4.83; N 24.93. $C_{20}H_{16}N_6$. Calculated, %: C 70.59; H 4.71; N 24.71.

Compounds VIIb–VIIi (*general procedure*). A suspension of equimolar amounts of compound **VI** and the corresponding carboxylic acid hydrazide in alcohol was heated for 0.5–1 h under reflux. The mixture gradually became homogeneous, and then a solid precipitated. The mixture was cooled, and the precipitate was filtered off, washed with alcohol and water, and recrystallized from DMF.

***N′*-(1-Methyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-yl)-benzohydrazide (VIIb).** Yield 69%, mp 244–245°C. ^{1}H NMR spectrum, δ, ppm: 3.95 s (3H), 7.4–8.2 m (6H), 9.8–11.0 br.m (2H). Found, %: C 58.42; H 4.54; N 31.26. $C_{13}H_{12}N_6O$. Calculated, %: C 58.21; H 4.48; N 31.34.

***N′*-(1-Methyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-yl)-*p*-methoxybenzohydrazide (VIIc)**. Yield 78%, mp 226–228°C. ^{1}H NMR spectrum, δ, ppm: 3.8 s (3H), 3.9 s (3H), 7–8.5 m (6H), 11–11.2 d (1H), 12.2 br.s (1H). Found, %: C 56.48; H 4.91; N 28.31. $C_{14}H_{14}N_6O_2$. Calculated, %: C 56.37; H 4.70; N 28.19.

***N′*-(1-Benzyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-yl)-benzohydrazide (VIId).** Yield 73%, mp 229–230°C. ^{1}H NMR spectrum, δ, ppm: 5.5 s (2H), 7.2–8.0 m (10H), 8.12 s (1H), 8.38 s (1H), 11.5 d (1H), 12.5 br.s (1H). Found, %: C 66.41; H 4.72; N 24.36. $C_{19}H_{16}N_6O$. Calculated, %: C 66.28; H 4.65; N 24.42.

***N′*-(1-Benzyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-yl)-*p*-methoxybenzohydrazide (VIIe).** Yield 78%, mp 216–217°C. ^{1}H NMR spectrum, δ, ppm: 3.8 s (3H), 5.6 d (2H), 7.1–8.5 m (12H), 11.1 s (1H). Found, %: C 64.53; H 4.68; N 22.72. $C_{20}H_{18}N_6O_2$. Calculated, %: C 64.17; H 4.81; N 22.46.

***N′*-(1-Phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-yl)-*p*-methoxybenzohydrazide (VIIf).** Yield 69%, mp 231–233°C. ^{1}H NMR spectrum, δ, ppm: 3.9 s (3H), 7.0–8.6 m (11H), 9.8–11.6 br.m (2H). Found, %: C 63.27; H 4.51; N 23.15. $C_{19}H_{16}N_6O_2$. Calculated, %: C 63.34; H 4.45; N 23.33. *M* 360.

2-(4-Chlorophenoxy)-*N′*-(1-phenyl-1*H*-pyrazolo-[3,4-*d*]pyrimidin-4-yl)acetohydrazide (VIIg). Yield 86%, mp 217–218°C. ^{1}H NMR spectrum, δ, ppm: 5.5 s (2H), 6.8–8.5 m (11H), 11.5–11.7 br.d (2H). Found, %: C 60.72; H 3.56; Cl 9.62; N 22.46. $C_{19}H_{13}ClN_6O$. Calculated, %: C 60.56; H 3.45; Cl 9.43; N 22.31.

***N′*-(1-*p*-Tolyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-yl]-benzohydrazide (VIIh).** Yield 88.7%, mp 262–263°C. ^{1}H NMR spectrum, δ, ppm: 2.5 s (3H), 7.2–8.2 m (11H), 10.0–11.1 m (2H). Found, %: C 66.41; H 4.70; N 24.65. $C_{19}H_{16}N_6O$. Calculated, %: C 66.28; H 4.65; N 24.42. *M* 344.

4-Methyl-*N′*-(1-*p*-tolyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-yl)benzohydrazide (VIIi). Yield 89.5%, mp 273–274°C. ^{1}H NMR spectrum, δ, ppm: 2.4 s (6H), 7.3–8.4 m (10H), 9.8–10.9 m (2H). Found, %: C 67.00; H 5.23; N 23.61. $C_{20}H_{18}N_6O$. Calculated, %: C 67.04; H 5.03; N 24.46.

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