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Three Ways of Reactions of 5-(3,5-Dimethyl-1*H*-pyrazol-1-yl)-2-phenyl-1,3-oxazole-4-carbonitrile and Its Analogs with Nitrogen-containing Bases

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Abstract—Substituted 5-(3,5-dimethyl-1*H*-pyrazol-1-yl)-1,3-oxazole-4-carbonitrile differently react with nitrogen bases having different numbers of labile hydrogen atoms. Treatment of the title compounds with secondary amines or morpholine results in nucleophilic replacement of the pyrazolyl substituent at C^5, the ozaxole ring remaining unchanged. Their reactions with primary amines are accompanied by cleavage of the oxazole ring with formation of the corresponding enamino nitriles. Hydrazine hydrate acts in a similar way, but enehydrazino nitriles thus formed undergo fast cyclization to give new 4,5-diaminopyrazole derivatives. The latter can be converted into substituted pyrazolo[1,5-*a*]pyrimidines whose structure has been proved by X-ray analysis.

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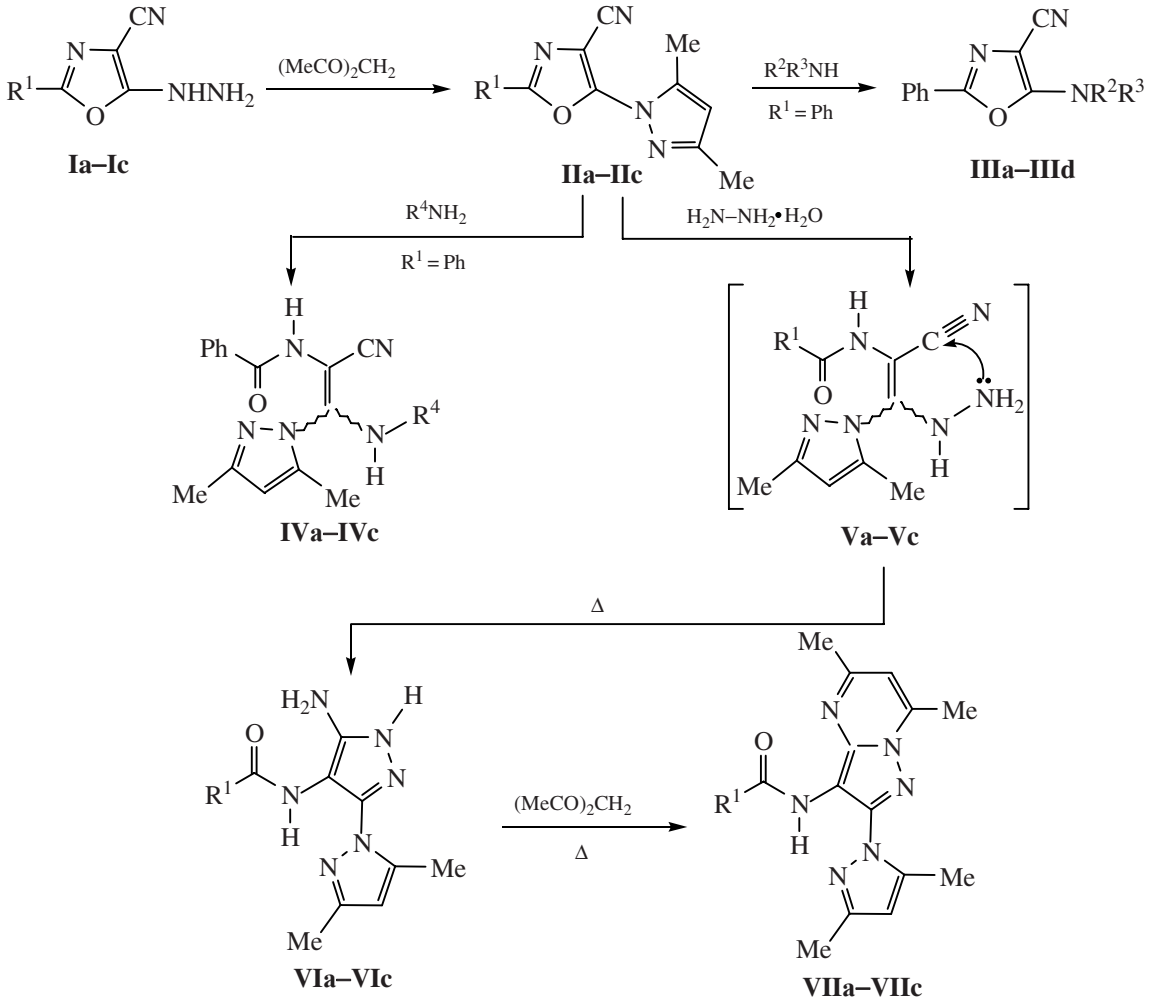
We previously showed that accessible 2-acylamino-3,3-dichloroacrylonitriles readily react with hydrazine hydrate to give the corresponding 2-alkyl(aryl, hetaryl)-5-hydrazino-1,3-oxazole-4-carbonitriles (**I**) [1]. Systematic studies on the reactivity of these biphilic compounds has just been started, but it is already clear that they are capable of undergoing unusual transformations.

The present article reports on new transformation pathways of compounds **I** upon successive treatment first with acetylacetone and then with various nitrogen-centered bases. As we recently showed [2], in the first step substituted 5-(pyrazol-1-yl)-1,3-oxsazole-4-carbonitriles **II** are formed. Molecules **II** possess several electrophilic centers, and we have found that they react with nitrogen bases having different numbers of labile hydrogen atoms along different pathways (Scheme 1).

Oxazole **II** (R^1 = Ph) readily reacted with dimethylamine, piperidine, and morpholine on heating, and the reaction involved nucleophilic replacement of the 3,5-dimethylpyrazolyl group on C^5 by the secondary amine residue due to electron-withdrawing effect of the cyano group on C^4. Here, the oxazole ring was conserved, and the product structure was confirmed by independent synthesis of compounds **III** by the known method, cyclocondensation of 2-benzoylamino-3,3-dichloroacrylonitrile with the corresponding amine [3, 4]. The transformation **II**→**III** attracts some interest from the theoretical viewpoint, for it provides an additional example of relatively rare nucleophilic substitution at C^5 in oxazoles [5]; however, from the preparative viewpoint, it is less efficient than the direct synthesis from chlorine-containing enamido nitriles like $Cl_2C{=}C(CN)\cdot NHCOR^1$ [6].

Interestingly, strongly basic primary amines, such as methylamine, benzylamine, and 2-phenylethylamine, reacted with substituted oxazoles **II** along quite different pathway. The reaction was accompanied by cleavage of the oxazole ring, elimination of the 3,5-dimethylpyrazolyl group did not occur, and the products were substituted enamino nitriles **IV**. The steric configuration of compounds **IV** was not determined, but the IR and ^1H and ^{13}C NMR spectra indicated predominant formation of only one geometric isomer. Analogous oxazole ring cleavage was observed previously in the reactions of 5-amino-4-

Scheme 1.



R^1 = Me (**a**), Ph (**b**), 2-thienyl (**c**); R^2R^3N = Me_2N (**a**), piperidino (**b**), morpholino (**c**), $H_2NN(Me)$ (**d**); R^4 = Me (**a**), $PhCH_2$ (**b**), $PhCH_2CH_2$ (**c**).

Table 1. Yields, melting points, and elemental analyses of compounds **I–VII**

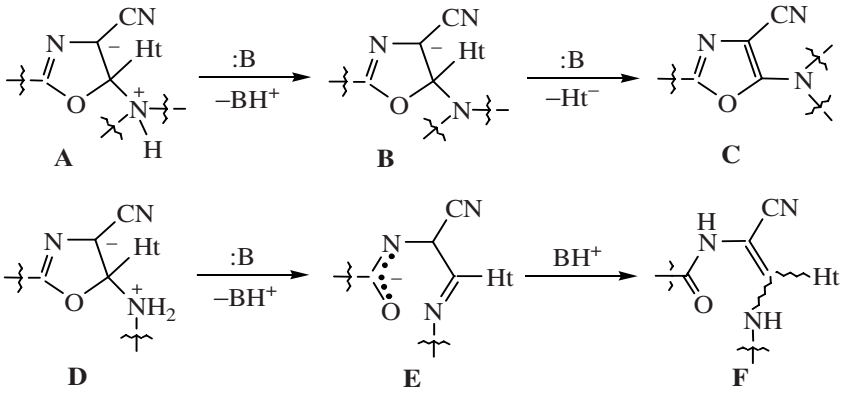
Comp. no.	Yield, %	mp, °C (from ethanol)	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
Ic	85	230 (decomp.)	46.87	2.63	27.51	$C_8H_6N_4OS$	46.59	2.93	27.17
IIa	60	169–170	59.64	4.90	27.58	$C_{10}H_{10}N_4O$	59.40	4.98	27.71
IIc	75	152–153	57.31	3.35	20.76	$C_{13}H_{10}N_4OS$	57.76	3.73	20.73
IIIa	50	146–147	67.77	5.26	19.84	$C_{12}H_{11}N_3O$	67.59	5.20	19.71
IIIb	60	126–127	71.18	5.85	16.26	$C_{15}H_{15}N_3O$	71.13	5.97	16.59
IIIc	65	131–132	65.56	5.16	16.19	$C_{14}H_{13}N_3O_2$	65.87	5.13	16.46
IIId	55	189–190	61.94	4.47	26.52	$C_{11}H_{10}N_4O$	61.67	4.71	26.15
IVa	55	197–198	65.32	5.41	23.46	$C_{16}H_{17}N_5O$	65.07	5.80	23.71

Table 1. (Contd.)

Comp. no.	Yield, %	mp, °C (from ethanol)	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
IVb	85	166–167	71.08	5.63	18.51	$C_{22}H_{21}N_5O$	71.14	5.70	18.85
IVc	75	186–187	71.49	6.36	18.19	$C_{23}H_{23}N_5O$	71.67	6.01	18.17
VIa	50	192–193	51.65	6.21	35.95	$C_{10}H_{14}N_6O$	51.27	6.02	35.87
VIb	90	145–146	60.54	5.37	28.70	$C_{15}H_{16}N_6O$	60.80	5.44	28.36
VIc	85	220–221	51.38	4.77	27.45	$C_{13}H_{14}N_6OS$	51.64	4.67	27.79
VIIa	90	227–228	60.43	6.19	28.09	$C_{15}H_{18}N_6O$	60.39	6.08	28.17
VIIb	95	186–187	66.53	5.49	23.32	$C_{20}H_{20}N_6O$	66.65	5.59	23.32
VIIc	90	210–212	59.19	5.01	22.85	$C_{18}H_{18}N_6OS$	59.00	4.95	22.93

trifluoroacetyl-1,3-oxazole derivatives with primary and secondary amines [7]. Thus primary and secondary amines react with 4-cyano-1,3-oxazole derivatives **II**, following different pathways. Primary adducts of oxazoles **II** with piperidine or morpholine contain a specific fragment **A** with one labile hydrogen atom. Elimination of that hydrogen atom as proton promotes the transformation **B** → **C**, which is favored by electron deficiency and appreciable nucleofugality of the dimethylpyrazolyl group (Scheme 2).

Scheme 2.



Ht = 3,5-dimethyl-1*H*-pyrazol-1-yl; B is a nitrogen base.

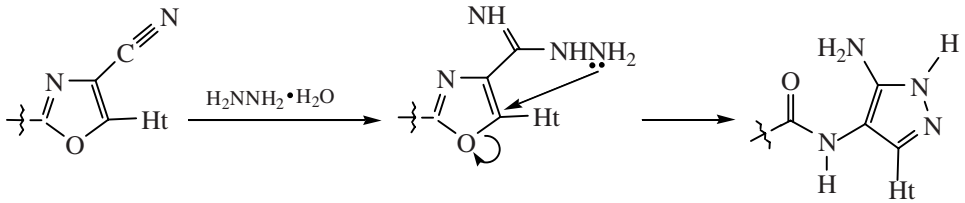
Adducts of oxazole **II** with primary amines (structure **D**) possess two labile hydrogen atoms, so that they could give rise to more diverse transformations. In particular, intermediate structure **E** having a C=N bond can be formed. This structure cannot arise from structure **A**. Probably, an important factor responsible for the transformations of structures **A** and **D** is steric hindrances at the C^5 atom, which affect mutual arrangement of the dimethylpyrazolyl substituent and the oxazole ring and hence the ability of the latter to undergo cleavage at the C–O or C–N bond.

Ring opening in substituted oxazoles **II** occurs in reactions with not only primary amines but also with hydrazine hydrates. Here, intermediate enehydrazino nitriles **V** cannot be isolated as individual substances, for they readily undergo intramolecular cyclization to bipyrazoles **VI** (Scheme 1). An alternative scheme of formation of compounds **VI** via addition of hydrazine at the cyano group in **II**, followed by cleavage of the oxazole ring (transformation sequence **G** → **H** → **J**) is shown below.

Table 2. IR and ^{1}H NMR spectra of compounds **I–VII**

Comp. no.	IR spectrum, ν, cm^{-1}	^{1}H NMR spectrum (DMSO-d_6), δ, ppm
Ic	2220 (C≡N), 3100–3300 (NH, $NH_{2\ as}$)	4.77 br.s (2H, NH_2), 7.11 m (1H$_{thiophene}$), 7.45 m (1H$_{thiophene}$), 7.66 m (1H$_{thiophene}$), 9.18 br.s (1H, NH)
IIa	2260 (C≡N)	2.26 s (3H, CH_3), 2.43 s (3H, CH_3) 2.55 s (3H, CH_3), 6.16 s (1H, C^4–H$_{pyrazole}$)
IIc	2255 (C≡N)	2.26 s (3H, CH_3), 2.55 s (3H, CH_3), 6.22 s (1H, C^4–H$_{pyrazole}$), 7.23 m (1H$_{thiophene}$), 7.88 m (1H$_{thiophene}$), 7.92 m (1H$_{thiophene}$)
IIId	2225 (C≡N), 3250-3350 ($NH_{2\ as}$)	3.29 s (3H, CH_3), 5.13 br.s (2H, NH_2), 7.44 m (3H$_{apom}$), 7.79 m (2H$_{arom}$)
IVa	1640 (NC=O)[a], 2220 (C≡N), 3100–3300 (NH_{as})	2.21 s (3H, CH_3), 2.38 s (3H, CH_3), 2.40 br.d (3H, CH_3), 6.01 s (1H, C^4–H$_{pyrazole}$), 7.19 br.q (1H, NH), 7.52 m (3H$_{arom}$), 7.97 m (2H$_{arom}$), 9.42 br.s (1H, NH)
IVb	1640 (NC=O)[a], 2220 (C≡N), 3100–3300 (NH_{as})	2.07 s (3H, CH_3), 2.22 s (3H, CH_3), 3.90 br.d (2H, CH_2), 5.92 s (1H, C^4–H$_{pyrazole}$), 7.30 m (8H$_{arom}$), 7.95 br.t (1H, NH), 8.01 m (2H$_{arom}$), 9.49 br.s (1H, NH)
IVc	1660 (NC=O)[a], 2210 (C≡N), 3150–3350 (NH_{as})	2.23 s (3H, CH_3), 2.32 s (3H, CH_3), 2.63 m (2H, CH_2), 2.84 m (2H, CH_2), 6.05 s (1H, C^4–H$_{pyrazole}$), 6.97 m (2H$_{arom}$), 7.17 m (3H$_{arom}$), 7.38 br.t (1H, NH), 7.55 m (3H$_{arom}$), 8.00 m (2H$_{arom}$), 9.44 br.s (1H, NH)
VIa	1650 (NC=O)[a], 3000–3400 (NH, $NH_{2\ as}$)	1.91 s (3H, CH_3), 2.21 s (3H, CH_3), 2.25 s (3H, CH_3), 4.82 br.s (2H, NH_2), 5.90 s (1H, C^4–H$_{pyrazole}$), 8.85 br.s (1H, NH), 11.53 br.s (1H, NH)
VIb	1640 (NC=O)[a], 2900–3400 (NH, $NH_{2\ as}$)	2.21 s (3H, CH_3), 2.38 s (3H, CH_3), 5.17 br.s (2H, NH_2), 7.52 m (3H$_{arom}$), 7.91 m (2H$_{arom}$), 9.92 br.s (1H, NH), 11.56 br.s (1H, NH)
VIc	1650 (NC=O)[a], 3000–3400 (NH, $NH_{2\ as}$)	2.20 s (3H, CH_3), 2.33 s (3H, CH_3), 5.07 br.s (2H, NH_2), 5.97 s (1H, C^4–H$_{pyrazole}$), 7.17 m (1H$_{thiophene}$), 7.76 m (2H$_{thiophene}$), 9.71 br.s (1H, NH), 11.63 br.s (1H, NH)
VIIa	1670 (NC=O), 3250–3350 (NH_{as})	1.94 s (3H, CH_3), 2.19 s (3H, CH_3), 2.32 s (3H, CH_3), 2.53 s (3H, CH_3), 2.66 s (3H, CH_3), 5.98 s (1H, C^4–H$_{pyrazole}$), 6.91 s (1H, C^5–H$_{pirim}$), 9.25 s (1H, NH)
VIIb	1650 (NC=O), 3200–3350 (NH_{as})	2.11 s (3H, CH_3), 2.39 s (3H, CH_3), 2.52 s (3H, CH_3), 2.69 s (3H, CH_3), 5.97 s (1H, C^4–H$_{pyrazole.}$), 6.93 s (1H, C^5–H$_{pirim}$), 7.50 m (3H$_{arom}$), 7.95 m (2H$_{arom}$), 9.81 br.s (1H, NH)
VIIc	1650 (NC=O), 3250–3400 (NH_{as})	2.11 s (3H, CH_3), 2.33 s (1H, CH_3), 2.52 s (1H, CH_3), 2.69 s (3H, CH_3), 6.06 s (1H, C^4–H$_{pyrazole}$), 7.02 s (1H, C^5–H$_{pirim}$), 7.18 m (1H$_{thiophene}$), 7.83 m (1H$_{thiophene}$), 8.02 m (1H$_{thiophene}$), 9.98 br.s (1H, NH)

[a] Band with a shoulder.

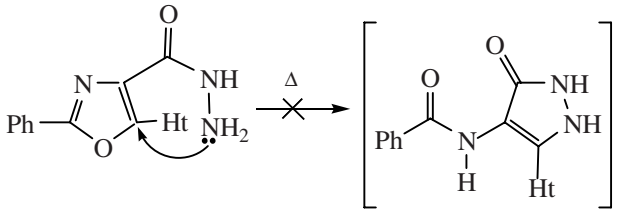


G **H** **J**

Ht = 3,5-dimethyl-1*H*-pyrazol-1-yl.

However, considerably lower electrophilicity of the C^5 atom in structure **H** compared to **G** should hinder the recyclization **H** → **J**. Moreover, we failed to effect analogous recyclization of compound **VIII** (**VIII** → **IX**) experimentally.

It should also be noted that no recyclization occurred in the reaction of oxazole **IIb** with methylhydrazine. In this case, nucleophilic replacement of the dimethylpyrazolyl group by $H_2NN(Me)$ substituent occurred. These data confirm the important role of



VIII **IX**

intermediate enehydrazino nitriles **V** in the formation of recyclization products **VI**. The structure of the latter is consistent with the IR spectral data, which showed the absence of cyano group in their molecules. The ^{1}H NMR spectra of **VI** contained proton signals assignable to a primary amino group, acylamino residue, and dimethylpyrazolyl fragment. In addition, the structure of compounds **VI** was proved by their reactions with acetylacetone, which lead to the formation of expected pyrazolo[1,5-*a*]pyrimidine derivatives **VII**. The structure of pyrazolo[1,5-*a*]pyrimidine **VIIb** (R^1 = Ph) was unambiguously determined by X-ray analysis (see figure).

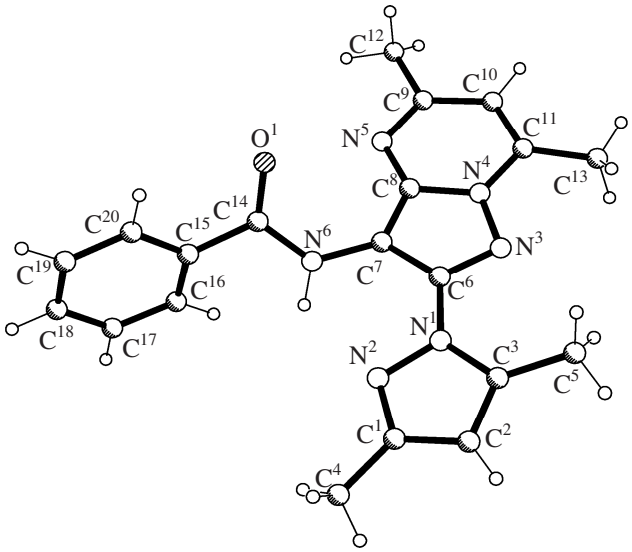
The central bicyclic system $N^1N^2C^6C^7C^8C^9C^{10}C^{11}$ in molecule **VIIb** is almost planar: the mean-square deviations of atoms from the corresponding plane is 0.0178 Å. The dimethyl-substituted pyrazole ring $N^1N^2C^1C^2C^3$ is turned through a dihedral angle of 11.3° with respect to the bicyclic fragment. The $N^6C^{14}C^{15}O^1$ plane with the $N^1N^2C^6C^7C^8C^9C^{10}C^{11}$ plane forms a dihedral angle of 50.7°. The N^6–C^4 bond [1.363(2) Å] is shorter than the standard single C–N bond (1.45 Å), and the sum of the bond angles at the N^6 atom is 356(1)°, indicating conjugation between the lone electron pair on N^6 and π system of the carbonyl group.

Thus rigorous determination of the structure of **VII** confirms the structure of products **VI** and the scheme of their formation by recyclization in the course of complex reaction of substituted oxazoles **II** with hydrazine hydrate.

EXPERIMENTAL

The IR spectra were recorded in KBr on a Specord M-80 spectrometer. The ^{1}H NMR spectra were measured on a Varian VRX-300 instrument from solutions in DMSO-d_6 using tetramethylsilane as internal reference.

The X-ray diffraction study on a 0.32×0.18×0.16-mm single crystal of compound **VIIb** was performed at room temperature on a Bruker Smart Apex II diffractometer (λMoK_α irradiation, graphite monochromator, θ_{max} = 26.41°, spherical segment $-9 \le h \le 9$, $-12 \le k \le 11$, $-12 \le l \le 15$). Total of 10132 reflections were measured, 3618 of which were independent (averaging factor R = 0.0305). Triclinic crystals, space group P-1 (no. 2); $C_{20}H_{20}N_6O$, M 360.42; a = 7.9965(3), b = 9.6792(4), c = 12.4874(5) Å; α = 81.561(3), β = 89.576(3), γ = 70.340(3)°; V = 899.41(6) Å^3; Z = 2, d_{calc} = 1.331 g cm^{-3}; μ = 0.087 mm^{-1}; F(000) = 380. The structure was solved by the direct method and was refined by the least-squares procedure in full-matrix anisotropic approximation using SHELXS-97 and SHELXL-97 software packages [8, 9]. The refinement was performed using 2291 reflections with $I > 2\sigma(I)$ (324 refined parameters, 7.07 reflections per parameter) and the weight scheme $\omega = 1/[\sigma^2(Fo^2) + (0.0518R)^2 + 0.0565R]$, where $R = (Fo^2 + 2Fc^2)/3$; the ratio of the maximal (average) shift to the error in the last iteration cycle was 0.011 (0.001). A correction for absorption was introduced using SADABS program (the ratio of the minimal and maximal corrections was



Structure of the molecule of *N*-[2-(3,5-dimethyl-1*H*-pyrazol-1-yl)-5,7-dimethylpyrazolo[1,5-*a*]pyrimidin-3-yl]benzamide (**VIIb**). Principal bond lengths (Å) and bond angles (deg): C^1–N^2 1.328(2), C^1–C^2 1.400(3), C^2–C^3 1.363(3), C^3–N^1 1.376(2), N^1–N^2 1.377(2), N^3–N^4 1.3597(19), C^6–N^3 1.338(2), C^6–C^7 1.399(2), C^7–C^8 1.391(2), C^8–N^5 1.353(2), C^8–N^4 1.386(2), C^9–N^5 1.321(2), C^9–C^{10} 1.419(3), C^{10}–C^{11} 1.357(3), C^{11}–N^4 1.368(2); $N^3C^6C^7$ 114.09(16), $N^3C^6N^1$ 118.64(15), $C^7C^6N^1$ 127.23(16), $C^8C^7N^6$ 127.92(16), $N^5C^8N^4$ 121.75(16), $N^5C^8C^7$ 132.72(17), $O^1C^{14}N^6$ 122.93(17), $N^2N^1C^6$ 117.81(14), $N^3N^4C^{11}$ 124.22(15).

T_{min}/T_{max} = 0.852338). All hydrogen atoms were visualized objectively from the Fourier difference series, and their positions were refined in isotropic approximation. The final divergence factors were $R_1(F^2)$ = 0.0826, $R_W(F^2)$ = 0.1148 (from all reflections, GOF 1.035) and $R_1(F)$ = 0.0451, $R_W(F^2)$ = 0.0982 [from reflections with $I > 2\sigma(I)$, GOF 1.035]. The residual electron density from the Fourier difference series after the last iteration cycle was 0.14 and –0.23 *e* Å^{-3}.

5-Hydrazino-2-methyl(phenyl)-1,3-oxazole-4-carbonitriles Ia and Ib were synthesized according to the procedure reported in [1]; 5-hydrazino-2-(2-thienyl)-1,3-oxazole-4-carbonitrile (**Ic**) was synthesized in a similar way. 5-(3,5-Dimethyl-1*H*-pyrazol-1-yl)-2-methyl(2-thienyl)-1,3-oxazole-4-carbonitriles **IIa–IIc** were prepared as described in [2].

5-Dimethylamino-2-phenyl-1,3-oxazole-4-carbonitrile (IIIa). A solution of 0.01 mol of compound **IIb** in 15 ml of ethanol was heated to the boiling point, and gaseous dimethylamine was passed through the solution over a period of 1 h. The mixture was then heated for 2 h under reflux, the solvent was removed under reduced pressure, the residue was treated with diethyl ether, and the precipitate was filtered off and recrystallized from ethanol. The product showed no depression of the melting point on mixing with an authentic sample of **IIIa** prepared as described in [3].

5-Piperidino- and 5-Morpholino-2-phenyl-1,3-oxazole-4-carbonitriles IIIb and IIIc (*general procedure*)**.** A solution of 0.01 mol of compound **IIb** in 15–20 ml of ethanol was heated to the boiling point, 0.05 mol of piperidine or morpholine was added dropwise, the mixture was heated for 3–5 h under reflux, the solvent was removed under reduced pressure, the residue was treated with diethyl ether, and the precipitate was filtered off and purified by recrystallization. The products showed no depression of the melting point on mixing with authentic samples of **IIIb** and **IIIc** prepared as described in [4].

5-(1-Methylhydrazino)-2-phenyl-1,3-oxazole-4-carbonitrile (IIId) was synthesized in a similar way from 5-(3,5-dimethyl-1*H*-pyrazol-1-yl)-2-phenyl-1,3-oxa-zole-4-carbonitrile (**IIb**) and methylhydrazine.

***N*-[2-(3,5-Dimethyl-1*H*-pyrazol-1-yl)-2-methylamino-1-cyanoethenyl]benzamide (IVa).** A solution of 0.01 mol of compound **IIb** in 15 ml of ethanol was heated to the boiling point, and gaseous methylamine was passed through the solution over a period of 1 h. The mixture was heated for 2 h under reflux, the solvent was removed under reduced pressure, the residue was treated with diethyl ether, and the precipitate was filtered off and recrystallized from ethanol.

***N*-[2-(R-Amino)-2-(3,5-dimethyl-1*H*-pyrazol-1-yl)-1-cyanoethenyl]benzamides IVb and IVc** (*general procedure*)**.** Compound **IIb**, 0.01 mol, was dissolved in 20 ml of ethanol, 0.05 mol of the corresponding amine was added, and the mixture was heated for 3–5 h under reflux. The solvent was removed under reduced pressure, the residue was treated with diethyl ether, and the precipitate was filtered off and purified by recrystallization.

3-Amino-4-acylamino-5-(3,5-dimethyl-1*H*-pyrazol-1-yl)pyrazoles VIa–VIc (*general procedure*)**.** Hydrazine hydrate, 0.05 mol, was added to a solution of 0.01 mol of compound **IIa–IIc** in 20 ml of ethanol, the mixture was heated for 5 h under reflux, the solvent was removed under reduced pressure, the residue was treated with diethyl ether, and the product was purified by recrystallization.

3-Acylamino-2-(3,5-dimethyl-1*H*-pyrazol-1-yl)-5,7-dimethylpyrazolo[1,5-*a*]pyrimidines VIIa–VIIc (*general procedure*)**.** Acetylacetone, 0.1 mol, was added to a suspension of 0.01 mol of compound **IVa–IVc** in 20 ml of toluene, and the mixture was heated for 5 h under reflux. Volatile substances were removed under reduced pressure, the residue was treated with diethyl ether, and the precipitate was filtered off and recrystallized from ethanol.

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