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# Recyclization of the Products of Amidine Addition to the Substituted 5-Amino-4-cyano-1,3-oxazole into New Derivatives of 5,6-Diaminopyrimidin-4-one

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**Abstract**—The products of addition of amidines to accessible 5-arylamino-4-cyano-1,3-oxazoles at heating in acetic acid undergo recyclization and afford new derivatives of 6-amino-5-acylaminopyrimidin-4-one, whose structure was reliably established using X-ray diffraction studies.

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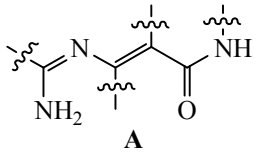
Derivatives of 5,6-diaminopyrimidin-4-one arise permanent interest in synthetic chemistry as biologically active substances and as intermediates in the synthesis of purine bases [1–7]. Despite the fact that chemical derivatives of 5,6-diaminopyrimidine have been well studied [8], some of these compounds are produced by a complex way, or are not known. Therefore the search for original, accessible initial substrates for the synthesis of new representatives of the 5,6-diaminopyrimidin-4-one derivatives is an important task.

In this paper we report on a method for obtaining previously unknown derivatives of 5,6-diaminopyrimidin-4-one with aryl substituents in positions 2 and 3 of the pyrimidine ring by recyclization of the products of addition of amidines to accessible 5-arylamino-4-cyano-1,3-oxazoles (**I**) [9] by heating them in acetic acid (Scheme 1). The initial stage of this process is the addition of amidines to the nitrile group of compounds **I**, which like with other nitrile [10, 11] proceeds with the formation of substrates **II.** The latter were isolated in individual state (Table 1). The structure of adducts **II** was reliably proved by the data of elemental analysis, mass spectra, and IR spectra, in the latter was lacking the band of stretching vibrations of the cyano group (Table 2). At the same time the comparison of $^{13}C$ NMR spectra of the compounds **Id** and **IIh** (Fig. 1) suggests that oxazole cycle remains intact at the **I** → **II** transformation.

The products **II** contain labile hydrogen atoms and therefore are able of prototropism with the formation of non-aromatic tautomers **III** containing unsaturated 5-imino azlactone system. At heating the latter in 98% acetic acid the azlactone system is cleaved by water, and thus further intramolecular **IV** → **V** cyclization is provided (Scheme 1).

Similar intramolecular cyclization with splitting off the ammonia molecule and the formation of the pyrimidine ring is known for a number of compounds with a characteristic fragment **A** [12, 13].

The structure of the final products of this complex




**A**

**II** → **V** transformation agrees with the mass spectra that contain a molecular peak greater by unity than for the substrates **II**. The elemental analysis data also indicate that formation of compounds **V** includes splitting off ammonia from the product **II** and adding a water molecule.

† Deceased.

**Table 1.** Yields, melting points, data of elemental analysis, and mass spectra of compounds **II** and **V**

| Comp. no. | Yield, % | mp, °C | Found, % | | | Formula | Calculated, % | | | $M^+$ |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | C | H | N | | C | H | N | |
| **IIa** | 52 | 167–170 | 72.97 | 5.27 | 17.65 | $C_{24}H_{21}N_5O$ | 72.89 | 5.35 | 17.71 | 395 |
| **IIb** | 81 | 200–202 | 73.24 | 5.73 | 17.19 | $C_{25}H_{23}N_5O$ | 73.33 | 5.66 | 17.10 | 409 |
| **IIc** | 78 | 197–199 | 60.86 | 4.30 | 14.74 | $C_{24}H_{20}BrN_5O$[a] | 60.77 | 4.25 | 14.76 | 473; 475 |
| **IId** | 68 | 203–205 | 60.83 | 4.32 | 14.82 | $C_{24}H_{20}BrN_5O$[b] | 60.77 | 4.25 | 14.76 | 473; 475 |
| **IIe** | 50 | 193–195 | 73.26 | 5.75 | 17.02 | $C_{25}H_{23}N_5O$ | 73.33 | 5.66 | 17.10 | 409 |
| **IIf** | 84 | 192–194 | 73.66 | 5.87 | 16.64 | $C_{26}H_{25}N_5O$ | 73.74 | 5.95 | 16.54 | 423 |
| **IIh** | 73 | 208–209 | 61.42 | 4.61 | 14.27 | $C_{25}H_{22}BrN_5O$[c] | 61.48 | 4.54 | 14.34 | 487; 489 |
| **IIh** | 64 | 138–140[d] | 68.35 | 5.66 | 21.09 | $C_{19}H_{19}N_5O$ | 68.45 | 5.74 | 21.01 | 333 |
| **Va** | 86 | 125–128 | 72.63 | 5.01 | 14.08 | $C_{24}H_{20}N_4O_2$ | 72.71 | 5.08 | 14.13 | 396 |
| **Vb** | 94 | 241–243 | 73.07 | 5.48 | 13.74 | $C_{25}H_{22}N_4O_2$ | 73.15 | 5.40 | 13.65 | 410 |
| **Vc** | 91 | 267–269 | 60.73 | 4.10 | 11.88 | $C_{24}H_{19}Br\ N_4O_2$[e] | 60.64 | 4.03 | 11.79 | 474; 476 |
| **Vd** | 81 | 284–286 | 60.57 | 3.96 | 11.69 | $C_{24}H_{19}Br\ N_4O_2$[f] | 60.64 | 4.03 | 11.79 | 474; 476 |
| **Ve** | 82 | 199–201 | 73.24 | 5.49 | 13.72 | $C_{25}H_{22}N_4O_2$ | 73.15 | 5.40 | 13.65 | 411 |
| **Vf** | 95 | 218–220 | 73.63 | 5.65 | 13.15 | $C_{26}H_{24}N_4O_2$ | 73.57 | 5.70 | 13.30 | 425 |
| **Vg** | 88 | 267–269 | 61.42 | 4.42 | 11.37 | $C_{25}H_{21}BrN_4O_2$[g] | 61.36 | 4.33 | 11.45 | 488; 490 |
| **Vh** | 70 | 291–293 | 68.34 | 5.49 | 16.72 | $C_{19}H_{18}N_4O_2$ | 68.25 | 5.43 | 16.76 | 335 |

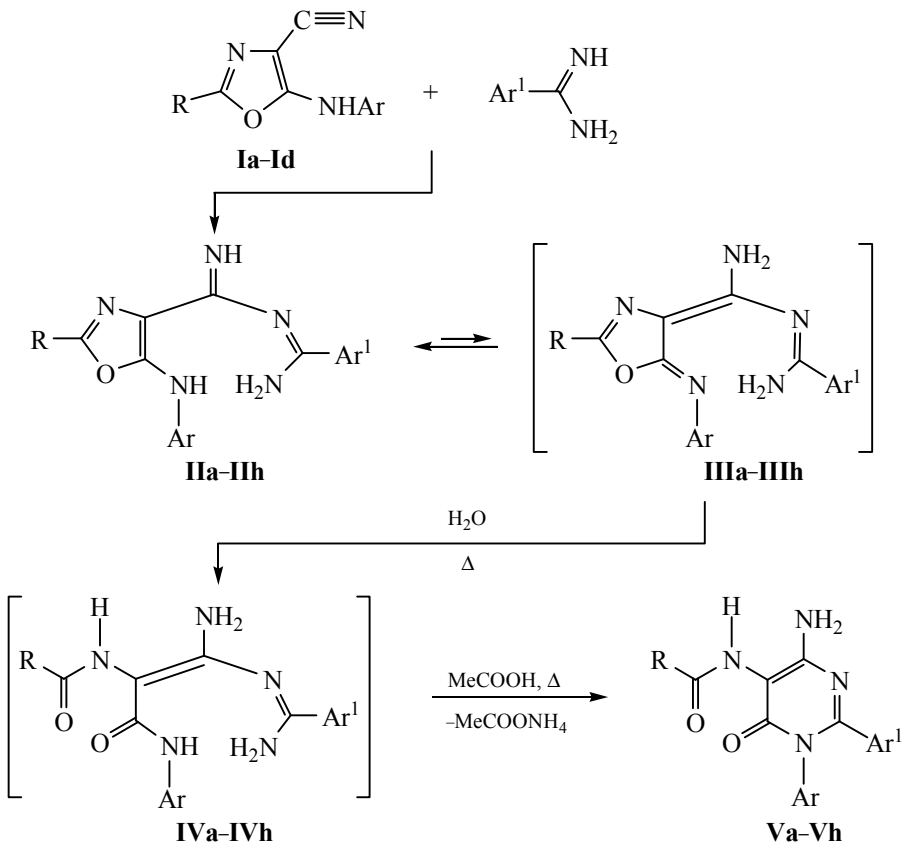
[a] Found, %: Br 16.93. Calculated, %: Br 16.84. [b] Found, %: Br 16.92. Calculated, %: Br 16.84. [c] Found, %: Br 16.28. Calculated, %: Br 16.36. [d] After recristallyzation from acetonitrile. [e] Found, %: Br 16.89. Calculated, %: Br 16.81. [f] Found, %: Br 16.73. Calculated, %: Br 16.81. [g] Found,%: Br 16.42. Calculated,%: Br 16.33.

**Table 2.** Spectral data of synthesized compounds

| Comp. no. | IR spectrum (KBr), ν, $cm^{-1}$ | $^1H$ NMR spectrum (DMSO-$d_6$), δ, mmp |
|---|---|---|
| **IIa** | 1676 (C=N)[a], 2200–2300 (bands not observed), 3050–3500 (NH as.) | 2.29 s (3H, $CH_3$), 7.09–7.93 m (14H arom.), 8.80 br.s (1H, NH)[b] |
| **IIc** | 1675 (C=N)[a], 2200–2300 (bands not observed), 2950–3430 (NH as.) | 2.27 s (3H, $CH_3$), 7.10–7.88 m (13H arom.), 8.79 br.s (1H, NH)[b] |
| **IIe** | 1681 (C=N)[a], 2200–2300 (bands not observed), 3000–3420 (NH as.) | 2.29 s (3H, $CH_3$), 2.35 s (3H, $CH_3$), 7.08–7.94 m (13H arom.), 8.78 br.s (1H, NH)[b] |
| **IIg** | 1677 (C=N)[a], 2200–2300 (bands not observed), 3000–3450 (NH as.) | 2.29 s (3H, $CH_3$), 2.35 s (3H, $CH_3$), 7.08–7.89 m (12H arom.), 8.74 br.s (1H, NH)[b] |
| **IIh** | 1674 (C=N)[a], 2200–2300 (bands not observed), 3020–3460 (NH as.) | 2.14 s (3H, $CH_3$), 2.24 s (3H, $CH_3$), 7.03–7.14 m (4H arom.), 7.46–7.94 m (5H arom.), 8.43 br.s (1H, NH)[b] |
| **Va** | 1642 (C=O)[a], 3010–3362 (NH as.) | 2.24 s (3H, $CH_3$), 6.49 br.s (2H, $NH_2$), 7.01–8.00 m (14H arom.), 9.16 s (1H, NH) |
| **Vb** | 1643 (C=O)[a], 3143–3397 (NH as.) | 2.23 s (3H, $CH_3$), 2.25 s (3H, $CH_3$), 6.46 br.s (2H, $NH_2$), 6.98–7.99 m (13H arom.), 9.15 s (1H, NH) |
| **Vc** | 1638 (C=O)[a], 3170–3311 (NH as.) | 2.27 s (3H, $CH_3$), 6.52 br.s (2H, $NH_2$), 7.02–7.52 m (13H arom.), 9.17 s (1H, NH) |
| **Vd** | 1650 (C=O)[a], 3049–3410 (NH as.) | 2.38 s (3H, $CH_3$), 6.52 br.s (2H, $NH_2$), 7.15–7.90 m (13H arom.), 9.08 s (1H, NH) |
| **Ve** | 1649 (C=O)[a], 3061–3297 (NH as.) | 2.23 s (3H, $CH_3$), 2.38 s (3H, $CH_3$), 6.59 br.s (2H, $NH_2$), 7.03–7.94 m (13H arom.), 9.15 s (1H, NH) |
| **Vf** | 1651 (C=O)[a], 2917–3355 (NH as.) | 2.23 s (3H, $CH_3$), 2.26 s (3H, $CH_3$), 2.38 s (3H, $CH_3$), 6.42 br.s (2H, $NH_2$), 6.98–7.90 m (12H arom.), 9.06 s (1H, NH) |
| **Vg** | 1632 (C=O)[a], 3040–3480 (NH as.) | 2.27 s (3H, $CH_3$), 2.38 s (3H, $CH_3$), 6.47 br.s (2H, $NH_2$), 7.02–7.90 m (12H arom.), 9.08 s (1H, NH) |
| **Vh** | 1645 (C=O)[a], 2984–3421 (NH as.) | 1.98 s (3H, $CH_3$), 2.22 s (3H, $CH_3$), 6.43 br.s (2H, $NH_2$), 7.00–7.28 m (9H arom.), 8.76 s (1H, NH) |

[a] Band with shoulder. [b] In exchange for NH, $NH_2$.

**Scheme 1.**




**I**: R = Ph, Ar = 4-$MeC_6H_4$ (**a**); R = 4-$MeC_6H_4$, Ar = Ph (**b**); R = 4-$MeC_6H_4$, Ar = 4-$MeC_6H_4$ (**c**); R = Me, Ar = 4-$MeC_6H_4$ (**d**); **II–V**: R = $Ar^1$ = Ph, Ar = 4-$MeC_6H_4$ (**a**); R = Ph, Ar = $Ar^1$ = 4-$MeC_6H_4$ (**b**); R = Ph, Ar = 4-$MeC_6H_4$, $Ar^1$ = 4-$BrC_6H_4$ (**c**); R = 4-$MeC_6H_4$, Ar = Ph, $Ar^1$ = 4-$BrC_6H_4$ (**d**); R = Ar = 4-$MeC_6H_4$, $Ar^1$ = Ph (**e**); R = Ar = $Ar^1$ = 4-$MeC_6H_4$ (**f**); R = Ar = 4-$MeC_6H_4$, $Ar^1$ = 4-$BrC_6H_4$ (**g**); R = Me, Ar = 4-$MeC_6H_4$, $Ar^1$ = Ph (**h**).

The $^1$H NMR spectra of compounds **V** contain the signals of the amide and the primary amino group protons in typical positions (9.06–9.16 and 6.42–6.52 ppm, respectively). Furthermore, the comparison of the $^{13}$C NMR spectra of compounds **IIh** and **Vh** indicates the transformation of oxazole ring (cf. the signals of carbon atoms of the methyl group in Fig. 1).

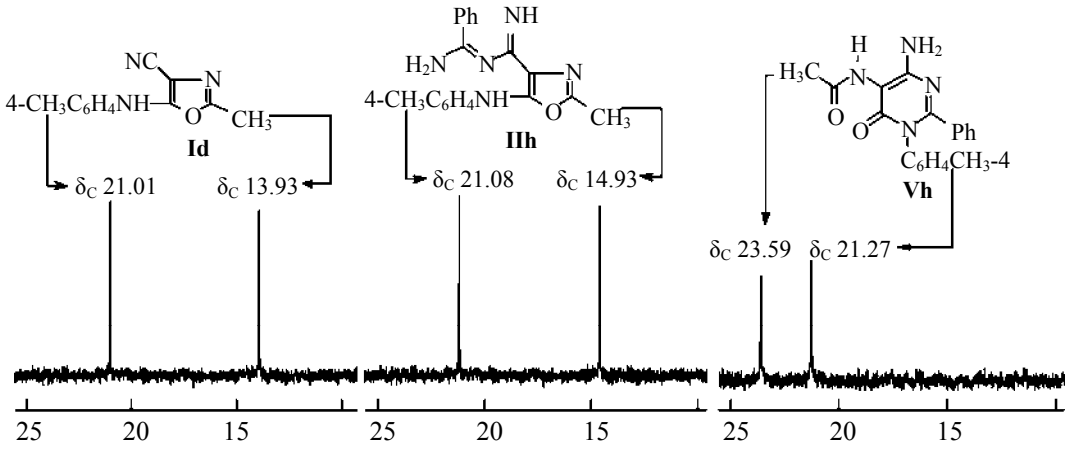



**Fig. 1.** Fragments of $^{13}$C NMR spectra of compounds **Id**, **IIh,** and **Vh**.

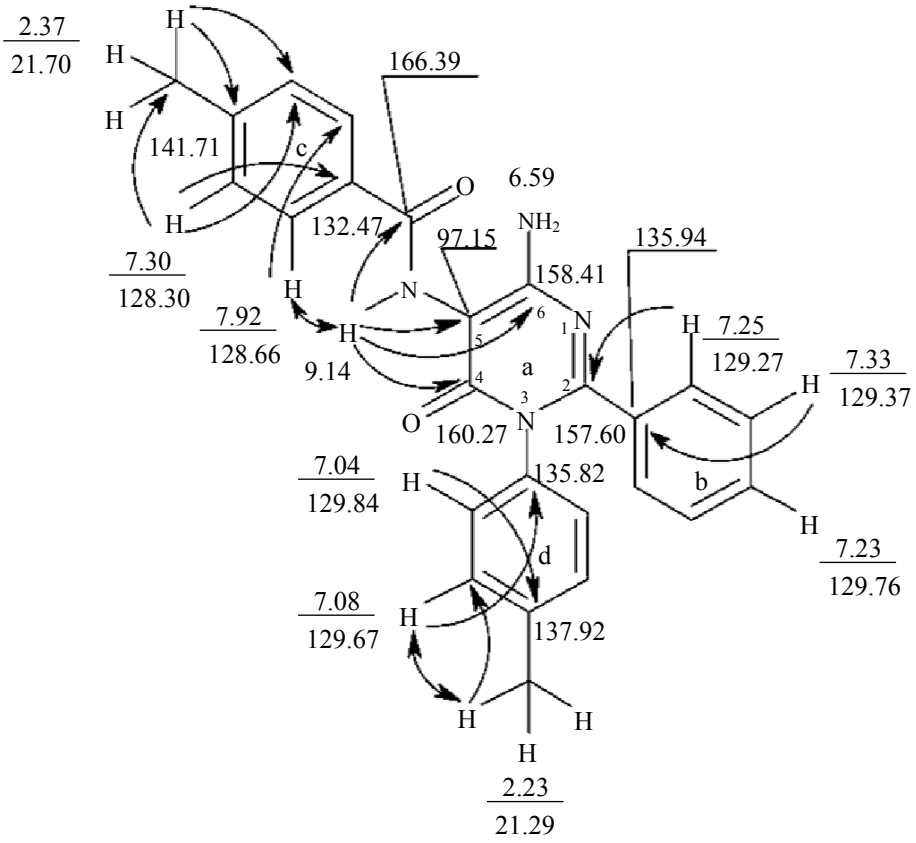



**Fig. 2.** The main correlations (shown by arrows) and the assignment of signals (ppm) in the $^{1}H$ and $^{13}C$ NMR spectra of compound **Ve**.

An important confirmation of the structure of compounds **V** was also obtained by a comprehensive analysis using two-dimensional NMR spectroscopy, which included homonuclear experiments COSY and NOESY, and heteronuclear HMBC and HMQC. Basic correlations for compound **Ve** are shown in Fig. 2, and all the found correlations are listed in Table 3. The two-dimensional HMBC spectrum revealed the interaction of the CONH proton (9.14 ppm) with the carbon nuclei CONH (166.39 ppm), $C^{5a}$ (97.15 ppm), $C^{4a}$ (160.27 ppm) and $C^{6a}$ (158.41 ppm), proving the cleavage of the oxazole cycle.

But all these data are still not sufficient to exclude other alternative structures. Therefore, for the unequivocal proof of the structure of compounds **V,** one of them (R = Ar = 4-$MeC_6H_4$, $Ar^1$ = Ph) was used for the X-ray diffraction analysis (Fig. 3). The distribution of bond lengths and angles in the pyrimidine cycle has no special features and is characteristic of such systems (Fig. 3). The heterocycle is almost planar, the mean deviation of atoms from the mean plane is 0.016 Å. Phenyl groups $C^{13}$–$C^{18}$ and $C^{20}$–$C^{25}$ at the heterocycle are turned by 82° and 72° respectively. The bonds $C^2N^3$ 1.352(3) and $C^5N^4$ 1.350(3) Å are significantly shorter compared with the standard [14] length of a single C–N bond (average 1.45 Å), which is associated with conjugation of the lone electron pair of nitrogen atoms $N^3$ and $N^4$ with the π-system of the heterocycle and the carbonyl bond, respectively.

The compound **Ve** crystallizes with one molecule of water. In the crystal a formation of the hydrogen bonds $O^3$–$N^{31O}$···$O^1$ and $N^3$–$H^{31N}$···$N^2$ is observed with the following parameters: $O^3H^{31O}$ 0.83(4), $O^3$···$O^1$ 2.9545(18) Å, $O^3$–$H^{31O}$–$O^1$ 143(4 )°, and $N^3$–$H^{31N}$ 0.90(3), $N^3$···$N^2$ 3.038(3) Å, $N^3$–$H^{31N}$–$N^2$ 173(2)°.

Since the intermediate compounds **IV** were not identified, it is possible that the synthesis of compounds **V** can also proceed along the path **II** → **VI** → **VII** → **V** (Scheme 2). A key role in this transformation play compounds **VII**, which are unstable in acidic medium and can be easily split with water like other oxazole[5,4-*d*]pyrimidines [15,16], but we believe that the preferable way of the formation of compounds **V** is the one shown in Scheme 1.

**Table 3.** The list of correlations in the COSY, NOESY, HMQC, and HMBC spectra of compound **Ve**[a]

| $^1$H, δ | $^1$H, δ | | $^{13}$C, δ($C^{3b}$H, $C^{5b}$H) | |
|---|---|---|---|---|
| | COSY | NOESY | HMQC | HMBC |
| 7.23 ($C^{4b}$H) | 7.33 | 7.33 | 129.76 | 129.27 ($C^{2b}$, $C^{6b}$) |
| 7.33 ($C^{3b}$H, $C^{5b}$H) | 7.23, 7.25 | 7.23, 7.25 | 129.37 | 129.37 ($C^{3b}$, $C^{5b}$), 135.94 ($C^{1b}$) |
| 7.25 ($C^{2b}$H, $C^{6b}$H) | 7.33 | 7.33 | 129.27 | 129.27 ($C^{2b}$, $C^{6b}$), 157.60 ($C^{2a}$), 129.76 ($C^{4b}$) |
| 6.59 ($NH_2$) | – | – | – | – |
| 2.37 ($CH_3C^{4c}$) | – | 7.30 | 21.70 | 141.71 ($C^{4c}$), 128.30 ($C^{3c}$, $C^{5c}$) |
| 7.30 ($C^{3c}$H, $C^{5c}$H) | 7.92 | 7.92, 2.37 | 128.30 | 128.30 ($C^{3c}$, $C^{5c}$), 21.70 ($\underline{C}H_3C^{4c}$), 132.47 ($C^{1c}$) |
| 7.92 ($C^{2c}$H, $C^{6c}$H) | 7.30 | 7.30, 9.14 | 128.66 | 128.66 ($C^{2c}$, $C^{6c}$), 166.39 (CONH) |
| 9.14 (CONH) | – | 7.92 | – | 166.39 (CONH), 97.15 ($C^{5a}$), 160.27 ($C^{4a}$), 158.41 ($C^{6a}$) |
| 2.23 ($CH_3C^{4d}$) | – | 7.08 | 21.29 | 129.67 ($C^{3d}$, $C^{5d}$), 137.92 ($C^{4d}$) |
| 7.08 ($C^{3d}$H, $C^{5d}$H) | 7.04 | 2.23, 7.04 | 129.67 | 135.82 ($C^{1d}$), 21.29 ($\underline{C}H_3C^{4d}$), 129.67 ($C^{3d}$,$C^{5d}$) |
| 7.04 ($C^{2d}$H, $C^{6d}$H) | 7.08 | 7.08 | 129.84 | 137.92 ($C^{4d}$), 129.84 ($C^{2d}$, $C^{6d}$) |

[a] For the numbering of carbon atoms see Fig. 2.

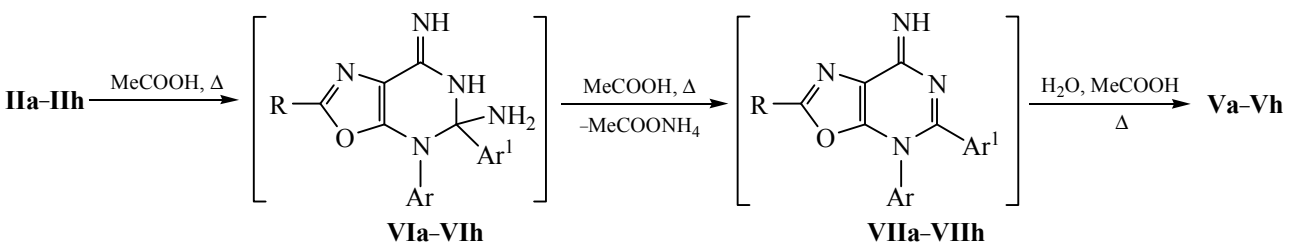
In conclusion, we note that the transformation of compounds **I** → **V** is of interest not only as an unusual transformation of the oxazole ring into the pyrimidine one, but also as a preparative method of synthesis of previously unknown derivatives of 5,6-diaminopyrimidin-4-one, the intermediates in the synthesis of purine bases. Their possible use in this direction will be considered by us in the future.

## EXPERIMENTAL

The IR spectra of the compounds were recorded on a Vertex 70 spectrometer from tablets with KBr, the $^1$H NMR spectra were taken on a Varian-300 instrument, and the $^1$H and $^{13}$C NMR spectra of compounds **Id, IIh, Vh,** and **Ve**, on a Varian Mercury-400 instrument in DMSO-$d_6$ with internal reference TMS. The mass spectra of compounds were recorded on an Agilent 1100/DAD/MSD VL G1965 instrument. The melting points were measured on a Fisher–Johns unit.

The X-ray diffraction study of a single crystal of compound **Ve** with linear dimensions 0.04×0.08×0.46 mm was carried out at room temperature on a Bruker Smart Apex II diffractometer (λMo$K_\alpha$ radiation, graphite monochromator, $\theta_{max}$ 26.40°, sphere segment $-15 \le h \le 8$, $-7 \le k \le 7$, $-36 \le l \le 37$). Overall number of measured reflections 12,733, of which 4.573 were independent (*R*-factor of averaging 0.0532). Crystals of compound **Ve**: $C_{25}H_{23}N_4O_{2.5}$, $M$ = 419.47, are monoclinic, space group $P2/c$, $a$ = 12.0149(7), $b$ = 6.1862(4), $c$ = 30.3489(17) Å, β = 97.266(3)°, $V$ = 2237.6(2 ) Å$^3$, $Z$ = 4, $d_{calc}$ = 1.245, μ = 0.082 mm$^{-1}$, $F$(000) = 884. The structure was solved by the direct method and refined by full-matrix least-mean- squares method in an anisotropic approximation using the

**Scheme 2.**




**VIa–VIh** **VIIa–VIIh**

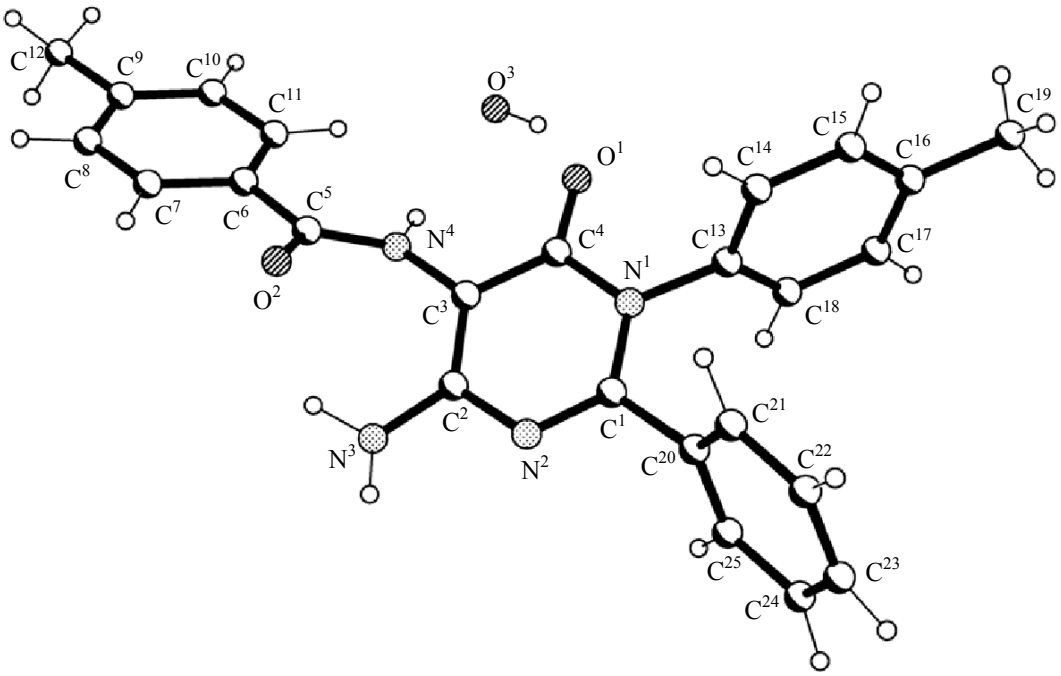



**Fig. 3.** General view of the molecule **Ve** and the main bond lengths and angles.

programs SHELXS97 and SHELXL97 [17, 18]. For the refinement 4573 reflections were used with $I > 2\sigma(I)$, (355 refined parameters, number of reflections per a parameter 12.9, the weight scheme was used, $\omega = 1/[\sigma^2(Fo^2) + (0.0598P)^2]$, where $P = (Fo^2 + 2Fc^2)/3$, the ratio of maximum (average) shift to the error in final cycle was 0.10(0.005). The extinction correction was introduced using the program SADABS (ratio of minimum to maximum corrections $T_{min}/T_{max}$ = 0.656381). The hydrogen atoms of methyl groups were localized geometrically, other hydrogen atoms were identified and refined isotropically. The final values of divergence factors are as follows: $R1(F)$ = 0.0531, $wR2(F^2)$ = 0.1085, $GOF$ = 0.953, over the reflections with $I > 2\sigma(I)$. Residual electron density obtained from the difference Fourier series after the last cycle of refinement was 0.18 and –0.18 $e/Å^3$.

A complete set of X-ray data for compound **Ve** is deposited in the Cambridge Structural Database (CCDC 742460).

***N*-[(2-Aryl-5-arylamino-1,3-oxazol-4-yl)(imino)-methyl]arylamidines (IIa–IIh)**. To a solution of 0.04 mol of oxazole **Ia–Id** [9] in 10 ml of anhydrous ethanol was added 0.08 mol of arylamidine, the mixture was refluxes for 4 h, the separated orange precipitate was filtered off, washed with ethanol and water, and a compound **IIa–IIh** formed was purified by recrystallization from ethanol.

**6-Amino-5-acylamino-2,3-diaryl-3*H*-pyrimidin-4-ones (Va–Vh).** A solution of 0.04 mol of a compound **IIa–IIh** in 20 ml of 98% acetic acid was refluxed for 2 h, 150 ml of water was then added, the precipitate formed was filtered off, and compound **Va–Vh** formed was crystallized from ethanol–water 9:1 mixture.

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