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Synthesis of 4-Alkyl-2-aryl-1,3-oxazole[5,4-*d*]pyrimidine-7(4*H*)-thiones and 6-Alkyl-2-aryl-1,3-oxazole[5,4-*d*]pyrimidin-7(6*H*)-ones from 2-Aroylamino-3,3-dichloroacrylonitriles

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Abstract—The sequential treatment of accessible 2-aroylamino-3,3-dichloroacrylonitriles with excess of amine, triethyl orthoformate, and hydrogen sulfide results in 4-substituted oxazole[5,4-*d*]pyrimidine-7(4*H*)-thione. The sequential reactions of the same reagents with sodium methylate, trifluoroacetic acid, triethyl orthoformate, and amines give rise to the 6-substituted oxazole[5,4-*d*]pyrimidin-7(4*H*)-one.

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It has been shown that 2-aroyl-3,3-dichloroacrylonitriles **I** and their analogs are valuable reagents for the heterocyclization leading to a number of mononuclear derivatives and condensed heterocycles [1–8].

In the course of the systematic study of these cyclizations we have found now two simple transformation chains: **I** → **III** → **V** → **VII** and **I** → **II** → **IV** → **VI** → **VIII**, which make it possible to regioselectively attach the alkyl substituents to the pyrimidine nitrogen atoms. The end products are new 4-alkyl-2-aryl-1,3-oxazole[5,4-*d*]pyrimidine-7(4*H*)-thiones **VIIa**–**VIIh** and 6-alkyl-2-aryl-1,3-oxazole[5,4-*d*]pyrimidin-7-(6*H*)-ones **VIIIa**–**VIIIe**.

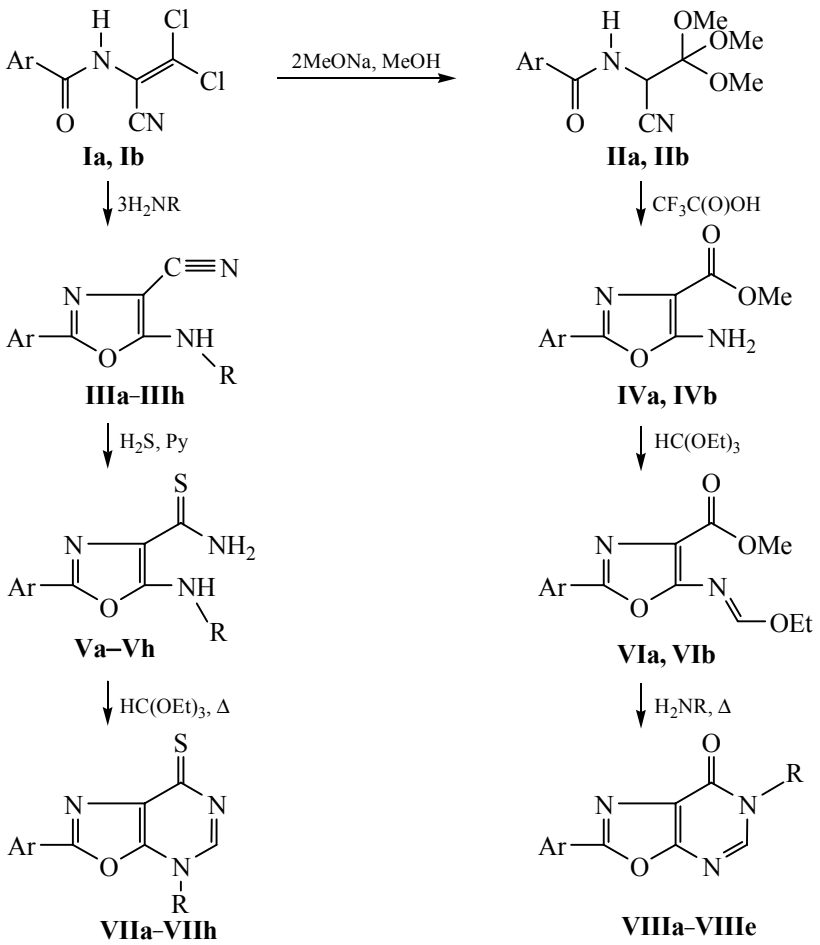
The first sequence of transformations involves the introduction of alkyl substituents to the N^4 atom of the oxazole[5,4-*d*]pyrimidine fragment already at the initial stage **I** → **III**, which has been studied previously [1]. The treatment of compounds **III** with excess hydrogen sulfide in pyridine yields thioamides **V**, which were used further in the cyclocondensation with triethyl orthoformate without additional purification, to form the final bicyclic products **VII**.

The yields, physicochemical constants, and elemental analysis data of the compounds obtained are given in Table 1.

To introduce alkyl substituents to the N^6 atom of oxazole[5,4-*d*]pyrimidine system we used 5-amino-2-aryl-4-metoxycarbonyl-1,3-oxazoles obtained earlier in the series of **I** → **II** → **IV** transformation [8, 9]. They were converted first into iminoesters **VI** by heating with an excess of triethyl orthoformate. The pyrimidine cyclization **VI** → **VIII** was carried out under the action of an excess of the corresponding amine.

All transformations represented in the scheme are in good agreement with the IR and ^{1}H NMR spectral data (Table 2). Thus, in the IR spectrum of the reaction mixture **III** → **V** the absorption bands of nitrile group at ν 2210–2220 cm^{-1} disappear. The ^{1}H NMR spectra contain a pair of broad singlets in the range of 8.30–8.70 ppm, which is characteristic of thioamide moiety. The formation of oxazolepyrimidines **VII** is accompanied with the disappearance in the IR spectra of the strong absorption band in the range of 2700–3500 cm^{-1}. Instead of the broadened signals of the amine moiety, the ^{1}H NMR spectra of compounds **VII** contain a singlet of the proton of the pyrimidine framework at δ 8.41–8.81 ppm, which is also present in the spectra of compounds **VIII**. In addition, the formation of pyrimidine ring in **V** → **VII** and **VI** → **VIII** transformations is in a good agreement with mass spectrometry data.

Although the chemistry of oxazole[5,4-*d*]pyrimidine derivatives has been studied quite well and there is a large number of publications on their synthesis and study of properties [10–13], the data on their 7-oxo-6-



I, II, IV, VI: Ar = Ph (**a**), 4-MeC_6H_4 (**b**); **III, V, VII**: Ar = Ph (**a–d**), 4-MeC_6H_4 (**e–h**); **VIII**: Ar = Ph (**a–c**), 4-MeC_6H_4 (**d, e**); R = $–(CH_2)_2OH$ (**IIIa, IIIe, Va, Ve, VIIa, VIIe, VIIIa, VIIId**), $–(CH_2)_3OH$ (**VIIIb**), $–(CH_2)_2O(CH_2)_2OH$ (**IIIb, IIIf, Vb, Vf, VIIb, VIIf**), $–CH_2COOH$ (**IIIc, IIIg, Vc, Vg, VIIc, VIIg**), $–(CH_2)_2COOH$ (**IIId, IIIh, Vd, Vh, VIId, VIIh**), **Bn (VIIIc, VIIIe**).

Table 1. Yields, melting points, and elemental analysis data of compounds **II–VIII**

Comp. no.	Yield, %	mp, °C (solvent for recrystallization)	Found, %				Formula	Calculated, %			
			C	H	N	S		C	H	N	S
IIb	58	118–119 (EtOH)	60.51	6.61	10.01	–	$C_{14}H_{18}N_2O_4$	60.42	6.52	10.07	–
IIIa	66	94–95 (EtOH)	62.94	4.92	18.27	–	$C_{12}H_{11}N_3O_2$	62.87	4.84	18.33	–
IIIb	59	55–56 (EtOH)	61.62	5.48	15.45	–	$C_{14}H_{15}N_3O_3$	61.53	5.53	15.38	–
IIIe	67	131–133 (EtOH)	64.26	5.47	17.35	–	$C_{13}H_{13}N_3O_2$	64.19	5.39	17.27	–
IIIf	60	83–85 (EtOH)	62.73	5.90	14.69	–	$C_{19}H_{17}N_3O_3$	62.71	5.96	14.62	–
IIIg	76	192–194 (EtOH)	60.62	4.39	16.22	–	$C_{13}H_{11}N_3O_3$	60.70	4.31	16.33	–
IIIh	78	175–177 (EtOH)	62.05	4.91	15.57	–	$C_{14}H_{13}N_3O_3$	61.99	4.83	15.49	–
IVb	62	233–234 (MeOH)	62.15	5.28	12.11	–	$C_{12}H_{12}N_2O_3$	62.06	5.21	12.06	–
Va	87	158–159 (EtOH)	54.81	5.06	15.90	12.01	$C_{12}H_{13}N_3O_2S$	54.74	4.98	15.96	12.18
Vb	93	115–116 (EtOH)	54.78	5.63	13.74	10.35	$C_{14}H_{17}N_3O_3S$	54.71	5.57	13.67	10.43
Ve	90	177–178 (EtOH)	56.23	5.37	15.22	11.64	$C_{13}H_{15}N_3O_2S$	56.30	5.45	15.15	11.56
Vf	94	131–133 (EtOH)	56.13	6.03	13.12	10.02	$C_{15}H_{19}N_3O_3S$	56.06	5.96	13.07	9.98
Vg	92	183–185 (EtOH)	53.65	4.56	14.49	11.10	$C_{13}H_{13}N_3O_3S$	53.60	4.50	14.42	11.01
Vh	90	192–194 (EtOH)	55.01	4.89	13.84	10.59	$C_{14}H_{15}N_3O_3S$	55.07	4.95	13.76	10.50

Table 1. (Contd.)

Comp. no.	Yield, %	mp, °C (solvent for recrystallization)	Found, %				Formula	Calculated, %			
			C	H	N	S		C	H	N	S
VIa	65	71–72 (benzene)	61.25	5.22	10.27	–	$C_{14}H_{14}N_2O_4$	61.31	5.14	10.21	–
VIb	67	92–93 (benzene)	62.54	5.63	9.81	–	$C_{15}H_{16}N_2O_4$	62.49	5.59	9.72	–
VIIa	62	182–183 (toluene)	57.20	4.13	15.44	11.82	$C_{13}H_{11}N_3O_2S$	57.13	4.06	15.37	11.73
VIIb	56	163–164 (toluene)	56.84	4.83	13.29	10.18	$C_{15}H_{15}N_3O_3S$	56.77	4.76	13.24	10.10
VIIc	64	220 (EtOH)	54.42	3.22	14.71	11.23	$C_{13}H_9N_3O_3S$	54.35	3.16	14.63	11.16
VIId	67	274–276 (EtOH)	55.90	3.74	13.91	10.72	$C_{14}H_{11}N_3O_3S$	55.81	3.68	13.95	10.64
VIIe	60	192–194 (toluene)	58.60	4.64	14.70	11.23	$C_{14}H_{13}N_3O_2S$	58.52	4.56	14.62	11.16
VIIf	55	152–153 (toluene)	57.91	5.25	12.76	9.75	$C_{16}H_{17}N_3O_3S$	57.99	5.17	12.68	9.68
VIIg	63	230–233 (EtOH)	55.89	3.74	13.89	10.71	$C_{14}H_{11}N_3O_3S$	55.81	3.68	13.95	10.64
VIIh	62	236–237 (EtOH)	57.20	4.21	13.25	10.11	$C_{15}H_{13}N_3O_3S$	57.13	4.16	13.32	10.17
VIIIa	61	169–171 (EtOH)	60.78	4.40	16.42	–	$C_{13}H_{11}N_3O_3$	60.70	4.31	16.33	–
VIIIb	63	165–166 (EtOH)	61.90	4.91	15.57	–	$C_{14}H_{13}N_3O_3$	61.99	4.83	15.49	–
VIIIc	70	218–220 (EtOH)	71.36	4.40	13.96	–	$C_{18}H_{13}N_3O_2$	71.28	4.32	13.85	–
VIIId	74	180–181 (EtOH)	61.93	4.91	15.56	–	$C_{14}H_{13}N_3O_3$	61.99	4.83	15.49	–
VIIIe	72	196–197 (EtOH)	71.84	4.68	13.32	–	$C_{19}H_{15}N_3O_2$	71.91	4.76	13.24	–

Table 2. Spectral characteristics of compounds **II–VIII**

Comp. no.	IR spectrum (KBr), ν, cm^{-1}	^1H NMR spectrum (DMSO-d_6), δ, ppm	Mass spectrum, *m*/*z*
IIb	2220 (CN), 3300–3400 (NH_{as})	2.38 s (3H, CH_3), 3.39 s (9H, $3CH_3O$), 5.56 d (1H, CH), 7.30–7.83 m ($4H_{arom}$), 9.19 d (1H, NH)	278
IIIa	1650[a], 2210 (CN), 2900–3600 (NH, OH_{as})	3.43 m (2H, CH_2), 3.63 m (2H, CH_2), 4.92 t (1H, OH), 7.50–7.90 m ($5H_{arom}$), 8.48 t (1H, NH)	229
IIIb	1650[a], 2215 (CN), 3150–3550 (NH, OH_{as})	3.50 m (6H, $3CH_2$), 3.63 m (2H, CH_2), 4.48 t (1H, OH), 7.43–7.79 m ($5H_{arom}$), 8.39 t (1H, NH)	273
IIIe	1650[a], 2215 (CN), 2900–3600 (NH, OH_{as})	2.35 s (3H, CH_3), 3.41 m (2H, CH_2), 3.60 m (2H, CH_2), 4.91 m (1H, OH), 7.30–7.70 m ($4H_{arom}$), 8.42 br.s (1H, NH)	243
IIIf	1645[a], 2220 (CN), 3150–3550 (NH, OH_{as})	2.35 s (3H, CH_3), 3.51 m (6H, $3CH_2$), 3.62 m (2H, CH_2), 4.61 t (1H, OH), 7.30–7.70 m ($4H_{arom}$), 8.46 br.s (1H, NH)	287
IIIg	1650[a], 2220 (CN), 3050–3500 (NH, OH_{as})	2.35 s (3H, CH_3), 4.10 m (2H, CH_2), 7.31–7.70 m ($4H_{arom}$), 8.72 t (1H, NH), 13.02 br.s (1H, OH)	257
IIIh	1645[a], 2220 (CN), 3100–3500 (NH, OH_{as})	2.35 s (3H, CH_3), 2.57 m (2H, CH_2), 3.55 m (2H, CH_2), 7.31–7.70 m ($4H_{arom}$), 8.66 t (1H, NH), 12.35 br.s (1H, OH)	271
IVb	1645[a], 1690 (C=O), 3180, 3250 (NH_2)	2.35 s (3H, CH_3), 3.74 s (3H, CH_3O), 7.30–7.70 m ($4H_{arom}$), 7.45 s (2H, NH_2)	232
Va	1630[b], 2870–3400 (NH, OH_{as})	3.61–3.65 m (4H, $2CH_2$), 4.99 br.s (1H, OH), 7.52–7.92 m ($5H_{arom}$), 8.34 s (1H, NH), 8.68 s (1H, NH), 8.88 m (1H, NH)	263
Vb	1640[b], 2870–3400 (NH, OH_{as})	3.52 m (4H, $2CH_2$), 3.69 m (4H, $2CH_2$), 7.52–7.93 m ($5H_{arom}$), 8.37 s (1H, NH), 8.71 s (1H, NH), 8.86 m (1H, NH)	307
Ve	1634[b], 2800–3500 (NH, OH_{as})	2.37 s (3H, CH_3), 3.58–3.65 m (4H, $2CH_2$), 5.00 br.s (1H, OH), 7.33–7.82 m ($4H_{arom}$), 8.29 s (1H, NH), 8.63 s (1H, NH), 8.86 m (1H, NH)	277
Vf	1643[b], 2600–3400 (NH, OH_{as})	2.37 s (3H, CH_3), 3.52 m (4H, $2CH_2$), 3.69 m (4H, $2CH_2$), 7.33–7.81 m ($4H_{arom}$), 8.32 s (1H, NH), 8.67 s (1H, NH), 8.85 m (1H, NH)	321
Vg	1632[b], 1729 (C=O), 2900–3400 (NH, OH_{as})	2.37 s (3H, CH_3), 4.32 d (2H, CH_2), 7.33–7.78 m ($4H_{arom}$), 8.40 s (1H, NH), 8.76 s (1H, NH), 8.87 t (1H, NH), 13.08 br.s (1H, OH)	291
Vh	1651[b], 1716 (C=O), 2800–3600 (NH, OH_{as})	2.37 s (3H, CH_3), 2.67 m (2H, CH_2), 3.74 m (2H, CH_2), 7.33–7.82 m ($4H_{arom}$), 8.32 s (1H, NH), 8.66 s (1H, NH), 8.87 m (1H, NH), 12.46 br.s (1H, OH)	305
VIa[c]	1620 (C=N), 1717 (C=O)	1.43 t (3H, CH_3), 3.92 s (3H, CH_3), 4.48 q (2H, CH_2), 7.43–8.06 m ($5H_{arom}$), 8.57 s (1H, CH)	274

Table 2. (Contd.)

Comp. no.	IR spectrum (KBr), ν, cm^{-1}	^1H NMR spectrum (DMSO-d_6), δ, ppm	Mass spectrum, *m*/*z*
VIb[c]	1627 (C=N), 1719 (C=O)	1.44 t (3H, CH_3), 2.39 s (3H, CH_3), 3.92 s (3H, CH_3), 4.48 q (2H, CH_2), 7.24–7.95 m (4H_{arom}), 8.56 s (1H, CH)	288
VIIa	1675[b] (C=N), 3050–3500 (OH_{as})	3.70 m (2H, CH_2), 4.12 m (2H, CH_2), 5.00 br.s (1H, OH), 7.58–8.04 m (5H_{arom}), 8.44 s (1H, C^2H_{pyrim})	273
VIIb	1685[b] (C=N), 3057–3417 (OH_{as})	3.47 s (4H, 2CH_2), 3.74 m (2H, CH_2), 4.24 m (2H, CH_2), 4.62 br.s (1H, OH), 7.57–8.03 m (5H_{arom}), 8.48 s (1H, C^2H_{pyrim})	317
VIIc	1665[b] (C=N), 1753 (COOH), 3000–3400 (OH_{as})	4.82 s (2H, CH_2), 7.59–8.05 m (H_{arom}), 8.57 s (1H, C^2H_{pyrim}), 12.52 br.s (1H, OH)	287
VIId	1650 (C=N), 1737 (COOH), 2990–3300 (OH_{as})	2.81 t (2H, CH_2), 4.25 t (2H, CH_2), 7.58–8.03 m (5H_{arom}), 8.56 s (1H, C^2H_{pyrim}), 12.51 br.s (1H, OH)	301
VIIe	1655[b] (C=N), 2930–3375 (OH_{as})	2.39 s (3H, CH_3), 3.69 m (2H, CH_2), 4.11 m (2H, CH_2), 5.00 br.s (1H, OH), 7.36–7.91 m (4H_{arom}), 8.41 s (1H, C^2H_{pyrim})	287
VIIf	1673[b] (C=N), 3044–3470 (OH_{as})	2.39 s (3H, CH_3), 3.47 m (4H, 2CH_2), 3.74 m (2H, CH_2), 4.23 m (2H, CH_2), 4.52 br.s (1H, OH), 7.37–7.92 (4H_{arom}) 8.47 s (1H, C^2H_{pyrim})	331
VIIg	1670[b] (C=N), 1735 (COOH), 3015–3450 (OH_{as})	2.36 s (3H, CH_3), 4.83 s (2H, CH_2), 7.36–7.95 m (4H_{arom}), 8.51 s (1H, C^2H_{pyrim}), 12.52 br.s (1H, OH)	301
VIIh	1665[b] (C=N), 1738 (COOH), 3025–3450 (OH_{as})	2.37 s (3H, CH_3), 2.80 t (2H, CH_2), 4.24 t (2H, CH_2), 7.38–7.93 m (4H_{arom}), 8.54 s (1H, C^2H_{pyrim}), 12.55 br.s (1H, OH)	315
VIIIa	1700[d] (C=O), 2950–3300 (OH_{as})	3.68 m (2H, CH_2), 4.13 m (2H, CH_2), 4.99 m (1H, OH), 7.59–8.10 m (4H_{arom}), 8.47 s (1H, C^2H_{pyrim})	257
VIIIb	1698[d] (C=O), 3090–3400 (OH_{as})	1.86 m (2H, CH_2), 3.47 m (2H, CH_2), 4.13 m (2H, CH_2), 4.66 m (1H, OH), 7.61–8.12 m (5H_{arom}), 8.56 s (1H, C^2H_{pyrim})	271
VIIIc	1696[d] (C=O)	5.30 s (2H, CH_2), 7.32–8.12 m (10H_{arom}), 8.84 s (1H, C^2H_{pyrim})	303
VIIId	1700[d] (C=O), 2900–3300 (OH_{as})	2.40 s (3H, CH_3), 3.68 m (2H, CH_2), 4.13 m (2H, CH_2), 4.95 m (1H, OH), 7.40–7.99 m (4H_{arom}), 8.45 s (1H, C^2H_{pyrim})	271
VIIIe	1700[d] (C=O)	2.41 s (3H, CH_3), 5.29 s (2H, CH_2), 7.38–8.01 m (9H_{arom}), 8.81 s (1H, C^2H_{pyrim})	317

[a] 5-Amino-1,3-oxazole moiety [1]. [b] Broad band. [c] In $CDCl_3$. [d] Overlapping signals of S=O and C=N groups.

R-substituted derivatives **VIII** are poor [14–16]. 7-Thioxo-4-R-derivatives **VII** are the first representatives of this structure as well as their unknown 7-oxo-4-R-substituted analogs, whose synthesis we plan to describe in subsequent publications.

Compounds **VII** and **VIII** are structural analogs of purine bases. Among such compounds substances were found possessing diverse biological activity [15–17]. An attention is drawn to the fact that using the developed approach to the synthesis of N^4- and N^6-substituted derivatives of oxazole[5,4-*d*]pyrimidine we have regioselectively attached the hydroxyalkyl groups to the nitrogen atoms, which are the components of acyclonucleosides with antiviral properties [18–23].

EXPERIMENTAL

The IR spectra were recorded on a Specord M-80 spectrophotometer from KBr pellets. The ^1H NMR spectra were obtained on a spectrometer VXR-300 in DMSO-d_6 relative to internal TMS. The mass spectra were recorded on an Aglient 1100/DAD/MSD VL G1965 instrument. The melting points were measured on a Fisher–Johns instrument.

N-(2,2,2-Trimethoxyethyl-1-cyano)benzamide **IIa** was prepared as previously described [9]. *N*-(2,2,2-Trimethoxyethyl-1-cyano)-4-methylbenzamide **IIb** was synthesized similarly to compound **IIa** starting from **Ib**.

5-Alkylamino-2-aryl-4-cyano-1,3-oxazoles (IIIc, IIIg). To a solution of 0.01 mol of compound **I** in 40 ml of tetrahydrofuran was added 0.035 mol of the corresponding amine. The mixture was kept for 12 h at 20–25°C. After the amine hydrochloride was filtered off, the filtrate was evaporated in a vacuum. The residue was mixed with 30–40 ml of water, and the precipitate was filtered off. Compounds **IIIa, IIIb, IIIe, IIIf** were purified by the recrystallization from ethanol.

***N*-(2-Aryl-4-cyano-1,3-oxazol-5-yl)glycine (IIIc, IIIg).** To 20 ml of 50% aqueous ethanol solution of

0.033 mol of sodium hydroxide were added 0.033 mol of glycine and 0.01 mol of dichloroacrylonitrile **I**. The mixture was stirred for 24 h at 20–25°C. When the solvent was removed in a vacuum to a half volume, the residue was acidified with hydrochloric acid to pH ~4–5. The precipitate was filtered off, and the compounds **IIIc, IIIg** were purified by the recrystallization. The physicochemical constants of compound **IIIc** coincide with the literature data [24].

***N*-(2-Aryl-4-cyano-1,3-oxazol-5-yl)-β-alanines (IIId, IIIh)** were obtained similarly to compounds **IIIc, IIIg** from the enamides **Ia** or **Ib** and β-alanine. The melting point of compound **IIId** corresponds to the published data [24].

Methyl 5-amino-2-phenyl-1,3-oxazole-4-car-boxylate (IVa) was prepared by a known method [8]. **Methyl 5-amino-2-(4-methylphenyl)-1,3-oxazole-4-carboxylate (IVb)** was obtained similarly from orthoester **IIb**.

5-Alkylamino-2-aryl-1,3-oxazole-4-carbothioamides (Va, Vb, Ve, Vf). To a solution of 0.005 mol of compound **IIIa**, **IIIb**, **IIIe** or **IIIf** in 10 ml of pyridine was added 1 ml of triethylamine. The reaction mixture was saturated with dry hydrogen sulfide for ~1 h and kept for 12 h at 20–25°C. Then 50 ml of water was added carefully with stirring, the mixture was acidified with dilute hydrochloric acid (1:1) to pH ~2. The precipitate was filtered off and the target compounds were purified by the recrystallization.

***N*-(2-Aryl-4-thiocarbamoyl-1,3-oxazol-5-yl)glycine (Vc, Vg)** and ***N*-(2-aryl-4-thiocarbamoyl-1,3-oxazol-5-yl)-β-alanine (Vd, Vh)** were obtained by a known method [24]. The physicochemical constants correspond to the published data [24].

Methyl 2-aryl-5-ethoxymethyleneimino-1,3-oxazole-4-carboxylates (VIa, VIb). A solution of 0.015 mol of aminooxazole **IVa** or **IVb** and 0.00001 mol of methyl 4-phenylsulfonic acid in 20 ml of triethyl orthoformate was heated on a boiling water bath for 8 h. The solvent was removed in a vacuum, the residue was treated with hexane, the precipitate was filtered off and recrystallized.

4-Alkyl-2-aryl-1,3-oxazole[5,4-*d*]pyrimidine-7(4*H*)-thiones (VIIa–VIIh). A solution of 0.002 mol of the corresponding thioamides **Va–Vh** in 15 ml of triethyl orthoformiate was refluxed for 3 h and cooled to 20–30°C. The precipitate was filtered off, washed with diethyl ether, and recrystallized.

6-Alkyl-2-aryl-1,3-oxazole[5,4-*d*]pyrimidin-7(6*H*)-ones (VIIIa–VIIIe). To a solution of 0.02 mol of compound **VIa** or **VIb** in 50 ml of anhydrous ethanol was added 0.022 mol of the corresponding amine. The mixture was heated for 4 h and cooled. The precipitate was filtered off and recrystallized.

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