

Reaction of Cyanamides with *N,N*-Binucleophiles

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Abstract—Reactions of 4,6-dimethylpyrimidin-2-yl- and aroylcyanamides with benzene-1,2-diamine, ethylenediamine, cyclohexane-1,2-diamine, and naphthalene-1,8-diamine leads to 1*H*-benzimidazol-2-amine, imidazolidin-2-imine, perhydrobenzimidazol-2-imine, and 1*H*-perimidin-2-amine derivatives, respectively.

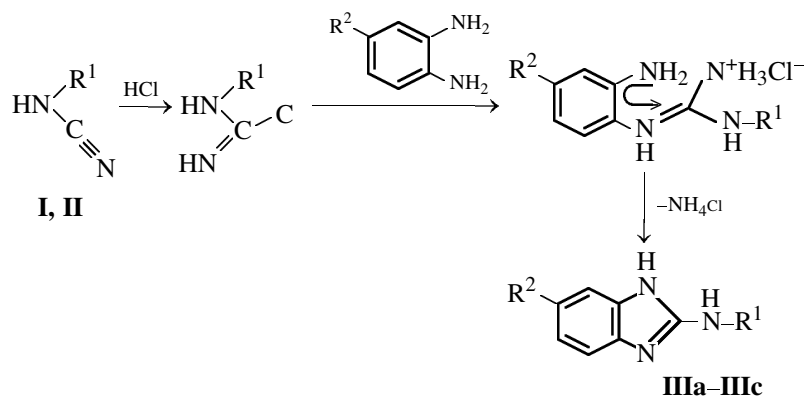
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Development of simple procedures for the synthesis of cyanamides and the possibility of using them for building up new heterocyclic systems underlie increased interest in these compounds [1, 2]. Cyanamides contain an electron-deficient carbon atom due to the presence of two neighboring nitrogen atoms; monosubstituted cyanamides can exist in the tautomeric $R-N=C=NH$ form, which makes them structurally similar to such reactive electrophiles as carbodiimides and isocyanates. Electron-acceptor substituents *R* enhance the reactivity of the cyanamide carbon atom toward nucleophiles, so that cyanamides can act as single-atom component in [*n* + 1] heterocyclizations with difunctional nucleophiles. We took advantage of this approach to synthesize heterocycles having an *N*–*C*–*N* fragment, namely imidazoles and pyrimidines.

o-Phenylenediamine is one of the most popular *N,N*-binucleophiles used in the synthesis of heterocyclic compounds. There are published data on the

preparation of benzimidazole derivatives from dialkyl- [3], acyl- [4], and some hetarylcyanoamides [5, 6]. In some cases, the heterocyclizations required acidic conditions. In order to make clear whether the presence of an acid catalyst is necessary, we used as substrates 4,6-dimethylpyrimidin-2-ylcyanamide (**I**) and 2-methoxyphenylcyanamide (**II**) with different electrophilicities of the cyanamide carbon atom. Both compounds almost did not react with *o*-phenylenediamine in the absence of HCl. The reactions in the presence of concentrated hydrochloric acid were complete in 3 h to give the corresponding benzimidazol-2-amine derivatives (Scheme 1). Presumably, hydrochloric acid enhances the electrophilicity of cyanamides via their transformation into chloroformamidines [2]; as a result, the reaction with aromatic amino group of the reagent becomes possible. Guanidine derivative formed in the first stage undergoes intramolecular ring closure with elimination of ammonia and formation of benzimidazole ring. Elimination of the guanidine nitrogen atom rather than of aromatic

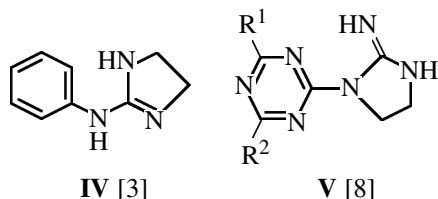
Scheme 1.



III, R¹ = 4,6-dimethylpyrimidin-2-yl, R² = H (**a**), Me (**b**), R¹ = 2-MeOC₆H₄, R² = H (**c**).

amino group is most probable. We previously showed [7] that reactions of disubstituted guanidines with isatoic anhydride involved the aromatic amino group as attacking nucleophile while the leaving group was guanidine fragment.

Another convenient *N,N*-binucleophile is ethylenediamine. Imidazole derivatives were obtained by reactions of ethylenediamine with dimethylcyanamide, phenylcyanamide [3], and triazinylcyanamides [8]. According to [3, 8], imidazol-2-amine derivatives with different positions of the substituent can be formed. The reaction with phenylcyanamide gives compound **IV** in which the phenyl group is attached to the exocyclic nitrogen atom; its structure was proved by independent synthesis. In this case, intermediate guanidine loses ammonia molecule with the nitrogen atom formerly belonging to the cyano group of the initial cyanamide. Structure **V** was proposed in [8] for the products obtained from ethylenediamine and triazinylcyanamide. According to the authors, acid properties of the NH group in 1,3,5-triazinylcyanamide favor formation of 2-aminoethylammonium salts in the reaction with ethylenediamine. These salts lose ammonia on heating, and new C–N bond is formed, followed by closure of imidazole ring.

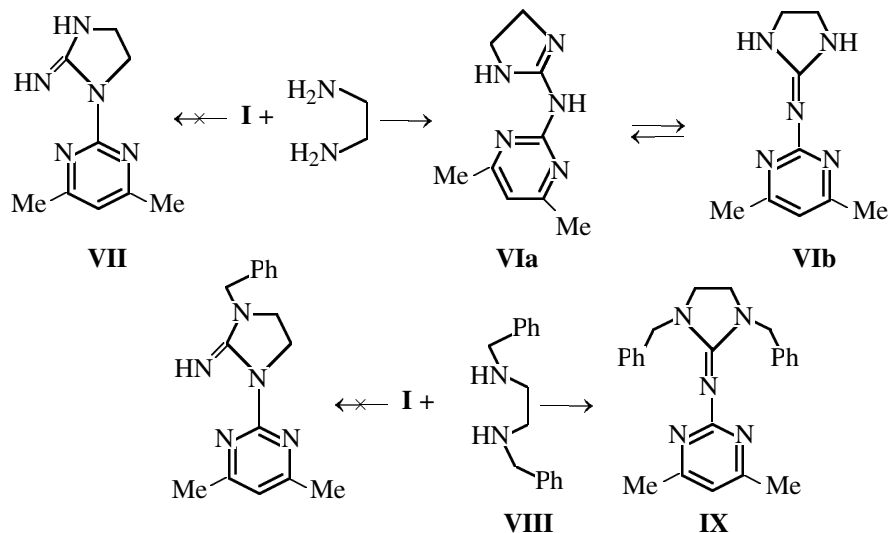


$R^1R^2 = \text{Alk(Ar)N}$; $R^1 = \text{AlkNH}$, $R^2 = \text{alkylamino}$, $R^2 = \text{MeO, MeS}$.

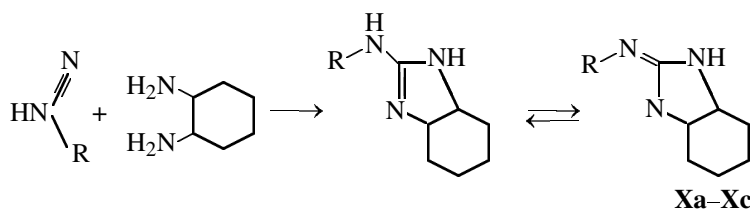
We synthesized imidazole derivative **VI** from 4,6-dimethylpyrimidin-2-ylcyanamide (**I**) and ethylenediamine (Scheme 2). The structure of compound **VI** was assigned on the basis of the following data. The ^1H NMR spectrum of the product contained two singlets at δ 8.3 and 4.52 ppm due to protons of the NH and CH_2 groups in the imidazole fragment. This pattern indicates magnetic equivalence of the above group, which is possible only for completely symmetric tautomeric structure **VIb**. In the mass spectrum of **VI** we observed peaks from fragment ions with m/z 123 and 67. These fragments could be formed via cleavage of the bond between the pyrimidine C^2 atom and exocyclic nitrogen atom. Analogous fragmentation of **VII** is impossible. Finally, the reaction of cyanamide **I** with *N,N'*-dibenzylethylenediamine (**VIII**) gave a compound which showed in the ^1H NMR spectrum signals from ten aromatic protons in the region δ 7.21–7.38 ppm and a singlet at δ 3.3 ppm from two benzylic CH_2 groups; only structure **IX** conforms to the observed spectral pattern (Scheme 2).

An alicyclic analog of ethylenediamine, *trans*-cyclohexane-1,2-diamine, also reacted with compound **I** and benzoylcyanamides (Scheme 3). As in the reactions with ethylenediamine, the products had the structure of tautomer **X** (according to the ^1H NMR data). The obtained imidazole derivatives may be regarded as cyclic guanidines, and they participate in reactions typical of these nucleophilic compounds. Compounds **VI** and **X** smoothly reacted with isocyanates and isothiocyanates to give the corresponding urea and thiourea derivatives **XI** and **XII** in good yields. As follows from the ^1H NMR spectra, the products are derivatives of tautomeric form **VIa**. By

Scheme 2.



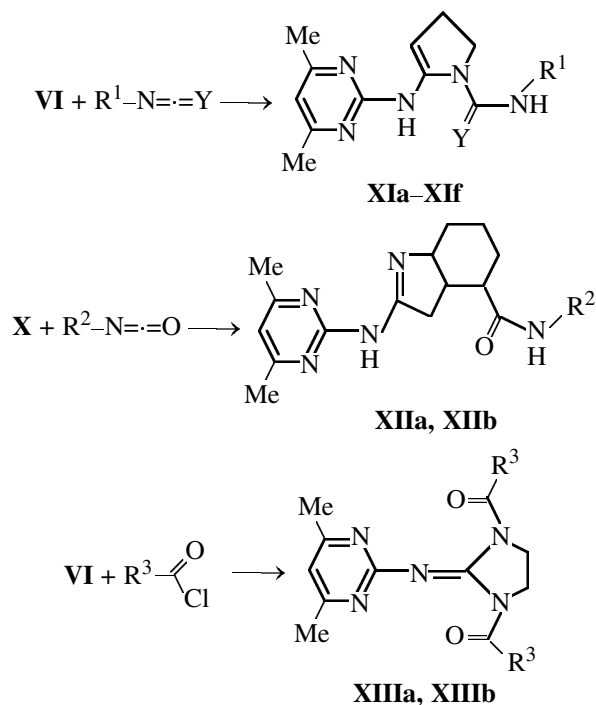
Scheme 3.



X, R = 4,6-dimethylpyrimidin-2-yl (a), PhCO (b), 4-ClC₆H₄CO (c).

reactions of **VI** with acyl chlorides we obtained stable 1,3-diacylimidazolidines **XIII** (Scheme 4).

Scheme 4.

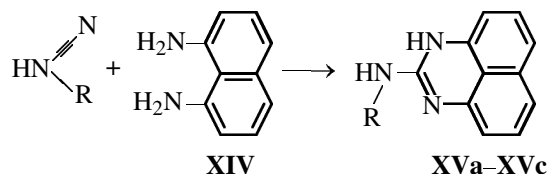


XI, Y = O; R¹ = Et (a), Ph (b), 3-ClC₆H₄ (c), 4-ClC₆H₄ (d); Y = S, R¹ = Ph (e), 3,4-Me₂C₆H₃ (f); **XII**, R² = Ph (a), 3,4-Cl₂C₆H₃ (b); **XIII**, R³ = Ph (a), 4-MeOC₆H₄ (b).

We expected formation of pyrimidine derivatives in reactions of cyanamides with nitrogen-centered 1,3-binucleophiles. In fact, naphthalene-1,8-diamine (**XIV**) reacted with pyrimidin-2-ylcyanamide **I** and benzoylcyanamides to afford 1H-perimidin-2-amine derivatives **XV** (Scheme 5).

The yields, melting points, and elemental analyses of the newly synthesized compounds are given in Table 1, and their ¹H NMR spectra are collected in Table 2. The prepared compounds attract interest as potential physiologically active substances.

Scheme 5.



X, R = 4,6-dimethylpyrimidin-2-yl (a), PhCO (b), 4-ClC₆H₄CO (c).

EXPERIMENTAL

The progress of reactions and the purity of products were monitored by TLC on Merck UV-254 plates using 15:5 and 20:1 chloroform-methanol mixtures as eluent. The ¹H NMR spectra were recorded on a Bruker AC-300 spectrometer (300 MHz) from solutions in DMSO-*d*₆ or DMSO-*d*₆-CCl₄; the chemical shifts were measured relative to tetramethylsilane. The mass spectra (electron impact, 70 eV) were obtained on an LKB-9000 instrument.

***N*-(4,6-Dimethylpyrimidin-2-yl)-1H-benzimidazol-2-amine (IIIa).** Concentrated hydrogen chloride, 0.93 ml, was added to a mixture of 1.48 g of cyanamide **I** and 1.08 g of *o*-phenylenediamine in 15 ml of propan-2-ol. The mixture was heated for 3 h under reflux and treated with a solution of 0.8 g of potassium hydroxide in 200 ml of distilled water. The precipitate was filtered off, washed with distilled water until neutral washings, and recrystallized from a mixture of *N,N*-dimethylacetamide (DMA)-2-propanol.

***N*-(4,6-Dimethylpyrimidin-2-yl)-5-methyl-1H-benzimidazol-2-amine (IIIb) and *N*-(2-methoxyphenyl)-1H-benzimidazol-2-amine (IIIc)** were synthesized in a similar way. Compound **IIIc** was recrystallized from propan-2-ol.

4,6-Dimethyl-*N*-(tetrahydro-1H-imidazol-2-ylidene)pyrimidin-2-amine (VI). Cyanamide **I**, 1.48 g, and ethylenediamine, 1.32 ml, were dissolved on heating in 15 ml of dioxane, and the mixture was

Table 1. Yields, melting points, and elemental analyses of compounds **III**, **VI**, **IX–XIII**, and **XV**

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
IIIa	47	290–291	65.35	5.32	29.43	C ₁₃ H ₁₃ N ₅	65.27	5.44	29.29
IIIb	32	312–313	66.25	5.83	27.65	C ₁₄ H ₁₅ N ₅	66.40	5.93	27.67
IIIc	56	162–164	70.34	5.31	17.62	C ₁₄ H ₁₃ N ₃ O	70.29	5.44	17.57
VI	47	225	56.67	6.88	36.56	C ₉ H ₁₃ N ₅	56.54	6.81	36.65
IX	61	125–127	74.27	6.65	18.94	C ₂₃ H ₂₅ N ₅	74.36	6.78	18.85
Xa	57	227–228	63.71	7.58	28.42	C ₁₃ H ₁₉ N ₅	63.67	7.76	28.57
Xb	36	270	69.01	7.09	17.28	C ₁₄ H ₁₇ N ₃ O	69.14	7.00	17.28
Xc	34	288–289	60.48	5.67	15.27	C ₁₄ H ₁₆ ClN ₃ O	60.48	5.76	15.12
XIa	63	157–158	54.85	6.82	32.14	C ₁₂ H ₁₈ N ₆ O	54.96	6.87	32.06
XIb	72	196	61.87	5.63	27.06	C ₁₆ H ₁₈ N ₆ O	61.94	5.81	27.10
XIc	80	197–198	55.63	4.86	24.22	C ₁₆ H ₁₇ ClN ₆ O	55.72	4.93	24.38
XId	70	222–223	55.84	4.85	24.27	C ₁₆ H ₁₇ ClN ₆ O	55.72	4.93	24.38
XIe	76	229–231	58.74	5.73	25.78	C ₁₆ H ₁₈ N ₆ S	58.90	5.52	25.77
XIf	61	201–203	61.02	6.38	23.60	C ₁₈ H ₂₂ N ₆ S	61.02	6.21	23.73
XIIa	68	241	66.05	6.60	23.06	C ₂₀ H ₂₄ N ₆ O	65.93	6.59	23.08
XIIb	65	232	55.40	5.13	19.28	C ₂₀ H ₂₂ Cl ₂ N ₆ O	55.43	5.08	19.40
XIIIa	18	260–262	69.10	5.32	17.36	C ₂₃ H ₂₁ N ₅ O ₂	69.17	5.26	17.54
XIIIb	24	285–286	65.30	5.31	15.34	C ₂₅ H ₂₅ N ₅ O ₄	65.36	5.45	15.25
XVa	49	264	70.70	5.12	24.24	C ₁₇ H ₁₅ N ₅	70.59	5.19	24.22
XVb	37	244–246	75.24	4.70	14.63	C ₁₈ H ₁₃ N ₃ O	75.26	4.53	14.63
XVc	24	258	67.04	3.85	12.88	C ₁₈ H ₁₂ N ₃ ClO	67.16	3.73	13.06

Table 2. ¹H NMR spectra of compounds **III**, **VI**, **IX–XIII**, and **XV**

Comp. no.	Chemical shifts δ, ppm
IIIa	2.51 s (6H, 2CH ₃), 6.75 s (1H, CH _{pyrim}), 7.05–7.22 m (2H, arom.), 7.32–7.44 m (2H, arom.), 11.38–11.52 (2H, NH)
IIIb	2.45 s (3H, CH ₃), 2.50 s (6H, 2CH ₃), 6.72 s (1H, CH _{pyrim}), 6.87 d (1H, arom.), 7.37–7.45 m (2H, arom.), 11.45–11.58 (2H, 2NH)
IIIc	4.05 s (3H, OCH ₃), 6.92–7.12 m (5H, arom.), 7.39 m (2H, arom.), 8.48 s (H, NH), 8.80 d (1H, arom.), 10.81 br.s (1H, NH)
VI	2.23 s (6H, 2CH ₃), 3.55 s (4H, CH ₂ CH ₂), 6.49 s (1H, CH _{pyrim}), 8.38 br.s (2H, 2NH)
IX	2.24 s (6H, 2CH ₃), 3.30 s (4H, CH ₂ CH ₂), 4.25 s (4H, 2CH ₂), 6.43 (1H, CH _{pyrim}), 7.24–7.35 m (10H, arom.)
Xa	1.39–1.52 m (4H, C ₄ H ₂ , C ₇ H ₂), 1.8–1.9 m (2H, CH ₂), 2.24 m (2H, CH ₂), 2.28 s (6H, 2CH ₃), 3.13 d (2H, 2CH), 6.43 s (1H, CH _{pyrim}), 8.92 br.s (2H, 2NH)
Xb	1.30–1.55 m (4H, C ₄ H ₂ , C ₇ H ₂), 1.78–1.90 m (2H, CH ₂), 2.09–2.11 m (2H, CH ₂), 3.07–3.18 m (2H, 2CH), 7.27–7.46 m (3H, arom.), 8.02–8.13 d (2H, arom.), 8.26 s (2H, 2NH)
Xc	1.30–1.47 m (4H, C ₄ H ₂ , C ₇ H ₂), 1.76–1.82 m (2H, CH ₂), 2.09–2.15 m (2H, CH ₂), 3.06–3.11 m (2H, 2CH), 7.39 d (2H, arom.), 8.05 d (2H, arom.), 8.30 s (2H, 2NH)
XIa	1.25 t (3H, CH ₂ CH ₃), 2.36 s (6H, 2CH ₃), 3.23 quint. (2H, NHCH ₂ CH ₃), 3.70 t (2H, CH ₂), 3.91 t (2H, CH ₂), 6.62 s (1H, CH _{pyrim}), 9.33 br.s (1H, NH), 10.04 t (H, NHCH ₂ CH ₃)
XIb	2.40 s (6H, 2CH ₃), 3.76 t (2H, CH ₂), 3.94 t (2H, CH ₂), 6.76 s (1H, CH _{pyrim}), 6.90–7.08 m (3H, arom.), 7.86–7.99 m (2H, arom.), 9.56 s (1H, NH), 12.87 s (1H, NH)
XIc	2.43 s (6H, 2CH ₃), 3.83 t (2H, CH ₂), 4.14 t (2H, CH ₂), 6.82 s (1H, CH _{pyrim}), 7.06–7.30 m (2H, arom.), 7.75 d (1H, arom.), 8.12 s (1H, arom.), 9.42 s (1H, NH), 13.02 s (1H, NH)

Table 2. (Contd.)

Comp. no.	Chemical shifts δ , ppm
XId	2.42 s (6H, 2CH ₃), 3.79 t (2H, CH ₂), 4.02 t (2H, CH ₂), 6.76 s (H, CH _{pyrim}), 7.29 d (2H, arom.), 7.60 d (2H, arom.), 9.42 s (H, NH), 13.02 s (H, NH)
XId	2.45 s (6H, 2CH ₃), 3.69 t (2H, CH ₂), 4.24 t (2H, CH ₂), 6.83 s (1H, CH _{pyrim}), 7.21–7.38 m (3H, arom.), 7.63–7.78 m (2H, arom.), 9.84 s (1H, NH), 14.76 s (1H, NH)
XIe	2.25 s (3H, CH ₃ arom.), 2.29 s (3H, CH ₃ arom.), 2.45 s (6H, 2CH _{3pyrim}), 3.71 t (2H, CH ₂), 4.32 t (2H, CH ₂), 6.70 s (1H, CH _{pyrim}), 7.08 d (1H, arom.), 7.47 m (2H, arom.), 9.61 s (1H, NH), 15.09 s (1H, NH)
XIIa	1.3–2.3 m (7H, aliph.), 2.44 s (6H, 2CH ₃), 2.9–3.5 m (3H, aliph.), 6.82 s (1H, CH _{pyrim}), 7.05–7.17 m (1H, arom.), 7.38 m (2H, arom.), 7.58 d (2H, arom.), 9.32 s (1H, NH _{guanid.}), 13.02 s (1H, NH _{amid})
XIIb	1.32–2.34 m (7H, aliph.), 2.45 s (6H, 2CH ₃), 2.9–3.4 m (3H, aliph.), 6.88 s (1H, CH _{pyrim}), 7.48 d (1H, arom.), 7.74 d (1H, arom.), 8.26 s (1H, arom.), 9.53 s (1H, NH _{guanid.}), 12.96 s (1H, NH _{amid})
XIIIa	2.05 s (6H, 2CH ₃), 4.02 s (4H, CH ₂ CH ₂), 6.47 s (1H, CH _{pyrim}), 7.28–7.59 m (10H, arom.)
XIIIb	2.08 s (6H, 2CH ₃), 3.80 s (6H, 2OCH ₃), 4.05 s (4H, CH ₂ CH ₂), 6.35 s (1H, CH _{pyrim}), 6.83 d (4H, arom.), 7.57 d (4H, arom.)
XVa	2.45 s (6H, 2CH ₃), 6.95 d (2H, naphth.), 7.12 s (1H, CH _{pyrim}), 7.35–7.53 m (4H, naphth.), 12.35 br.s. (2H, 2NH)
XVb	6.74 d (2H, naphth.), 7.11–7.28 m (4H, naphth.), 7.53–7.68 m (5H, arom.), 11.86 br.s. (2H, 2NH)
XVc	6.71 d (2H, naphth.), 7.11–7.28 m (4H, naphth.), 7.41 d (2H, arom.), 8.18 d (2H, arom.), 11.71 br.s. (2H, 2NH)

heated for 8 h under reflux. After cooling, the precipitate was filtered off and recrystallized from dioxane. Mass spectrum, m/z (I_{rel} , %): 191 (80), 190 (100), 162 (19), 149 (7), 134 (26), 123 (24), 108 (48), 93 (7), 67 (19), 55 (8), 42 (20).

***N*-(1,3-Dibenzyltetrahydro-1*H*-imidazol-2-ylidene)-4,6-dimethylpyrimidin-2-amine (IX).** A mixture of 1.48 g of cyanamide **I** and 2.88 g of *N,N*-dibenzylethylenediamine (**VIII**) in 15 ml of dioxane was heated for 10 h under reflux. After cooling, the precipitate was filtered off and recrystallized from dioxane.

4,6-Dimethyl-*N*-(perhydrobenzimidazol-2-ylidene)pyrimidin-2-amine (Xa). A mixture of 1.48 g of cyanamide **I** and 1.5 ml of trans-cyclohexane-1,2-diamine in 15 ml of dioxane was heated for 10 h under reflux. The mixture was cooled, and the precipitate was filtered off and recrystallized from dioxane.

***N*-(Perhydrobenzimidazol-2-ylidene)benzamide (Xb) and 4-chloro-*N*-(perhydrobenzimidazol-2-ylidene)benzamide (Xc)** were synthesized in a similar way.

2-(4,6-Dimethylpyrimidin-2-ylamino)-*N*-ethyl-4,5-dihydro-1*H*-imidazole-1-carboxamide (XIa). Compound **VI**, 1.91 g, was dissolved in 10 ml of anhydrous dioxane on heating, 0.8 ml of ethyl isocyanate was added, and the mixture was heated for 5 h at 90°C. The resulting solution was evaporated on a rotary evaporator, and the residue was recrystallized from 2-propanol.

2-(4,6-Dimethylpyrimidin-2-ylamino)-*N*-phenyl-4,5-dihydro-1*H*-imidazole-1-carboxamide (XIb). Compound **VI**, 1.91 g, was dissolved in 10 ml of anhydrous *N,N*-dimethylacetamide (DMA) on heating, 1.09 ml of phenyl isocyanate was added, and the mixture was heated for 5 h at 90°C. The precipitate was filtered off and recrystallized from dioxane–DMA.

***N*-(3-Chlorophenyl)-2-(4,6-dimethylpyrimidin-2-ylamino)-4,5-dihydro-1*H*-imidazole-1-carboxamide (XIc), *N*-(4-chlorophenyl)-2-(4,6-dimethylpyrimidin-2-ylamino)-4,5-dihydro-1*H*-imidazole-1-carboxamide (XId), 2-(4,6-dimethylpyrimidin-2-ylamino)-*N*-phenyl-4,5-dihydro-1*H*-imidazole-1-carbothioamide (XIe), and *N*-(3,4-dimethylphenyl)-2-(4,6-dimethylpyrimidin-2-ylamino)-4,5-dihydro-1*H*-imidazole-1-carbothioamide (XI_f)** were synthesized in a similar way.

2-(4,6-Dimethylpyrimidin-2-ylamino)-*N*-phenyl-3a,4,5,6,7,7a-hexahydro-1*H*-benzimidazole-1-carboxamide (XIIa). Compound **X**, 2.45 g, was dissolved in 10 ml of anhydrous DMA on heating, 1.09 ml of phenyl isocyanate was added, and the mixture heated for 5 h at 90°C. The precipitate was filtered off and recrystallized from dioxane–DMA.

***N*-(3,4-Dichlorophenyl)-2-(4,6-dimethylpyrimidin-2-ylamino)-3a,4,5,6,7,7a-hexahydro-1*H*-benzimidazole-1-carboxamide (XIIb)** was synthesized in a similar way.

3-Benzoyl-2-(4,6-dimethylpyrimidin-2-ylimino)-imidazolidin-1-yl(phenyl)methanone (XIIIa). Com-

pound **VI**, 0.96 g, was dissolved in 10 ml of anhydrous dioxane on heating, 1.5 ml of anhydrous triethylamine and 0.58 ml of benzoyl chloride were added in succession, and the mixture was heated for 2 h at 80°C. The precipitate of triethylamine hydrochloride was filtered off, the filtrate was evaporated on a rotary evaporator, and the residue was recrystallized from acetonitrile.

2-(4,6-Dimethylpyrimidin-2-ylimino)-3-(4-methoxybenzoyl)imidazolidin-1-yl(4-methoxyphenyl)methanone (XIIIb) was synthesized in a similar way.

N-(4,6-Dimethylpyrimidin-2-yl)-1H-perimidin-2-amine (XVa). Concentrated hydrochloric acid, 1.83 ml, was added to a mixture of 2.96 g of compound **I** and 3.16 g of naphthalene-1,8-diamine in 30 ml of propan-2-ol. The mixture was heated for 3 h under reflux and treated with a solution of 1.3 g of potassium hydroxide in 200 ml of distilled water. The precipitate was filtered off, washed with distilled water until neutral washings, and recrystallized from dioxane.

N-(1H-Perimidin-2-yl)benzamide (XVb). A mixture of 1.46 g of benzoylcyanamide and 1.58 g of naphthalene-1,8-diamine in 20 ml of dioxane was heated for 2 h at 90°C. The mixture was filtered, the filtrate was poured into 100 ml of 2-propanol, and the precipitate was filtered off.

4-Chloro-N-(1H-perimidin-2-yl)benzamide (XVc). A mixture of 1.81 g of 4-chlorobenzoylcyanamide and 1.58 g of naphthalene-1,8-diamine in 20 ml of dioxane was heated for 2 h at 90°C. The precipitate was filtered off and recrystallized from dioxane–DMA.

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