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Cascade Cyclization of 1,2-Diamino-4-phenylimidazole with Aromatic Aldehydes and Cyclohexanediones

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Abstract—The three-component condensation of 1,2-diamino-4-phenylimidazole with aromatic aldehydes and 1,3-cyclohexanediones occurred regioselectively and afforded 3-amino-1-phenyl-10-aryl-7,8-dihydroimidazo[1,5-*b*] cinnolin-9(5*H*,6*H*,10*H*)-ones.

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The presence in the molecule of 1,2-diamino-4-phenyl-imidazole of four nonequivalent nucleophilic sites provides alternative opportunities in the direction of the reactions with β-bielectrophilic carbonyl reagents, their synthetic precursor and equivalents and opens wide opportunities for the use of their imidazole system in the synthesis of diverse fused heterocyclic compounds. Primarily the vicinal diaminoazoles containing an N-amino group were considered in the reactions with enones as 1,4-binucleophiles leading to the formation of azolotriazepine derivatives [1, 2]. It was lately established that due to the lower nucleophilicity of amino groups in the 1,2-diaminoazoles than the nucleophilicity of the endocyclic reaction sites in the reactions with α,β-unsaturated ketones, their mono- and dibromo derivatives, with aroylacrylic acids, and also in the three-component condensations with aldehydes and Meldrum's acid formed not triazepine rings, but fused with azole ring pyridazine [3–6] and pyrimidine systems [7, 8].

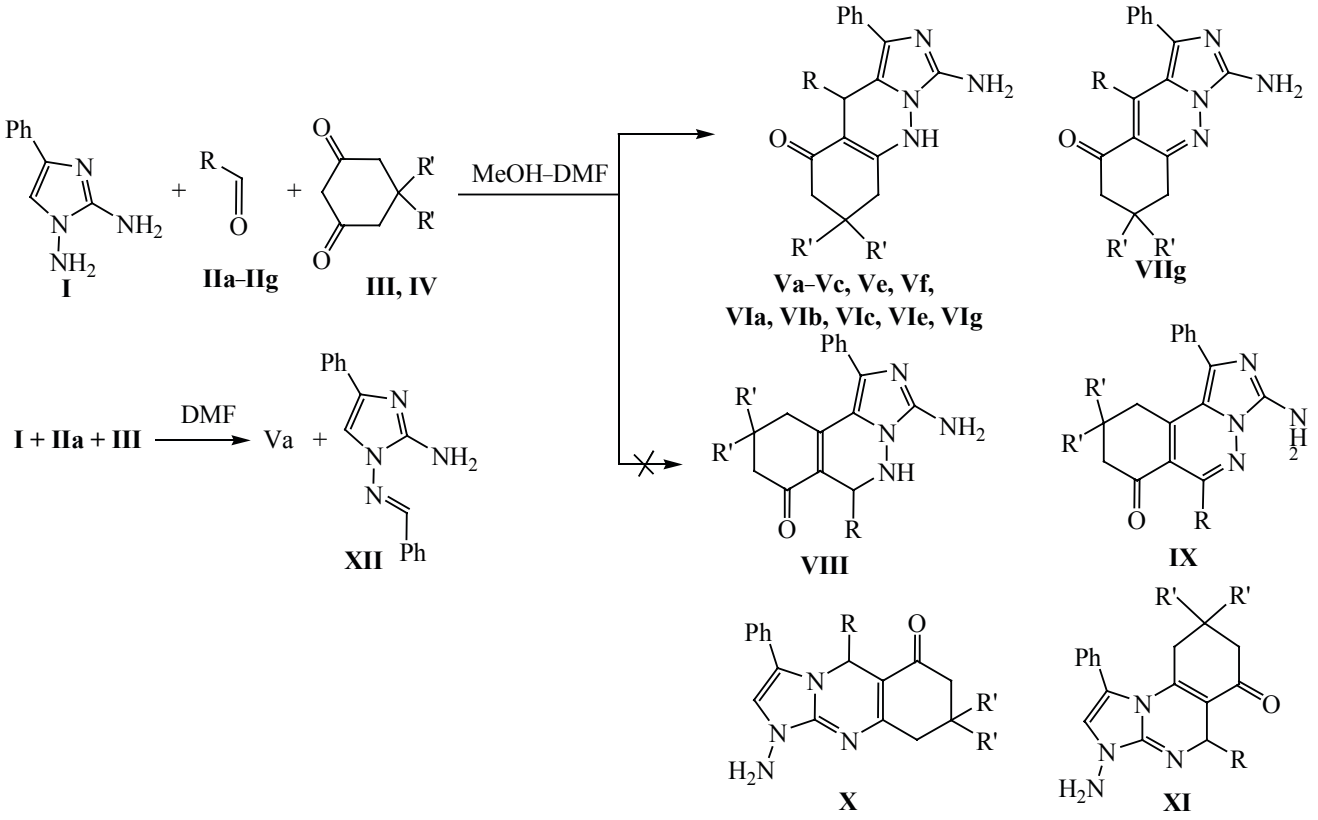
The target of this study was the establishment of the direction of the 1,2-diamino-4-phenylimidazole (**I**) reaction with aromatic aldehydes **IIa–IIg** and dimedone (**III**) or 1,3-cyclohexanedione (**IV**) under various conditions.

At boiling of equimolar amounts of amine **I** with benzaldehydes **IIa–IIg** and dimedone (**III**) or 1,3-cyclohexanedione (**IV**) both in methanol for 2 h or in DMF for 1 h we obtained dihydroimidazo[1,5-*b*] cinnolin-9-ones **Va–Vc, Ve, Vf** and **VIa, VIb, VId, VIe, VIg** respectively. In the three-component condensation involving *p*-nitrobenzaldehyde (**IIg**) and dimedone (**III**) in DMF compound **VIIg** formed within 3–5 min (see the scheme). Unlike virtually colorless crystalline compounds **V** and colorless amorphous powders **VI** nitro derivative **VIIg** is a dark-red crystalline substance.

Imidazocinnolinones of angular structure **VIII, IX** and/or quinazolinone systems **X, XI** were not detected in any of the experiments. In order to understand the reason of low yields of imidazocinnolinones **Va–Vc, Ve**, **Vf** it was shown by an example of the reaction of **I** with benzaldehyde (**IIa**) and dimedone (**III**) in DMF that in the reaction mixture alongside the target product **Va** formed 2-amino-4-phenyl-1-benzylideneaminoimidazole (**XII**). The presence of the same substance also in the reaction with 1,3-cyclohexanedione (**IV**) was confirmed by TLC. Evidently the corresponding azomethines of structure **XII** formed as side products in all reactions under consideration reducing the yields of imidazocinnolinones **V, VI**.

The structures of previously unknown compounds **Va–Vc, Ve, Vf** and **VIa, VIb, VId, VIe, VIg** were proved by IR and ^{1}H NMR spectra, their compositions were established from the elemental analysis, and azomethine **XII** was identified by the comparison its physicochemi-

Scheme.



II, V, VI, VII, R = Ph (a), 4-MeC_6H_4 (b), 4-$MeOC_6H_4$ (c), 4-$Me_2NC_6H_4$ (d), 4-ClC_6H_4 (e), 4-FC_6H_4 (f), 4-$NO_2C_6H_4$ (g); R' = Me (III, V, VII), H (V, VI).

cal and spectral characteristics with the published data [9, 10]. The structure of imidazocinnolinone **VIIg** was proved by XRD analysis (see the figure).

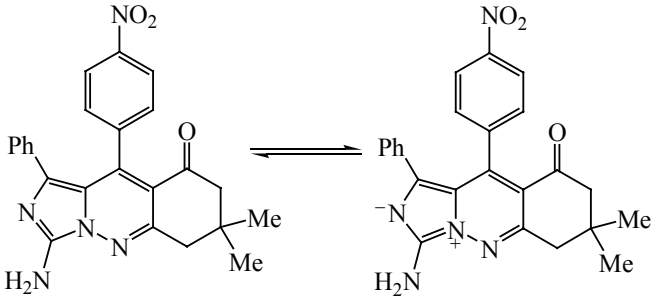
The appearance in the mass spectra of compounds **Va, VIa** of molecular ion peaks of *m*/*z* 384.54 and 356.46 respectively shows that these compounds form with a water molecule elimination. Yet the formation of structure **VIIg** beside the cyclocondensation required also a heteroaromatization, therefore the mass changed not by 18, but by 20 Da as compared to the mass of the initial reagents. Actually, the mass spectrum of imidazocinnolinone **VIIg** contained a peak M^+ of *m*/*z* 427.52 corresponding to this character of transformations leading to the formation of this compound.

In the IR spectra of all imidazo[1,5-*b*]cinnolin-9-ones **Va–Vc, Ve, Vf** and **VIa, VIb, VId, VIe, VIg** the most characteristic absorption bands originate from carbonyl groups (1688–1684 cm^{-1}), associated groups NH_2, NH, and methylene groups of the cyclohexenone ring in the region 3428–2660 cm^{-1}.

In the ^1H NMR spectra of compounds **Va–Vc, Ve, Vf** signals are observed indicating the presence in the structure of two aryl rings, one amino group (broadened singlet at δ 5.59–5.70 ppm), two methyl and two methylene groups of the cyclohexenone fragment (one of the CH_2 fragments appears as the *AB* system), and also of methane proton H^{10} as a singlet at δ 5.06–4.80 ppm. The signal of the NH group is not observed due to proton exchange. In the spectrum of imidazocinnolinone **VIIg** the signals of CH and NH protons of pyridazine ring are absent. The spectra of compounds **VI** are distinguished from those described above belonging to dimedone derivatives **V** by the lack of the signals from the methyl groups and the presence of three CH_2 fragments as multiplets in the strong field. ^1H NMR spectra make it possible to distinguish the structures of imidazocinnolinones **V, VI** with the linear junction of the rings from the isomeric compounds of angular structure **VIII**. In the case of the formation of the latter the spin-spin coupling should have arisen between the protons of the CHNH fragment that should be seen in the characteristic splitting of the corresponding

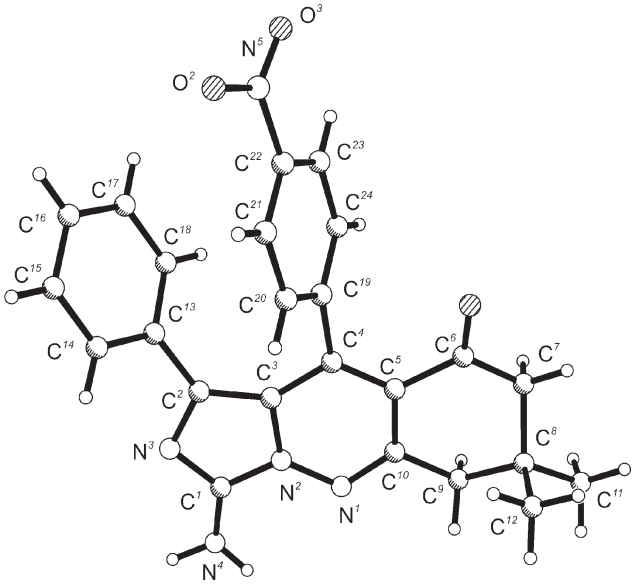
signals. Yet the obtained spectral data are not sufficient for distinguishing between the possible structures of imidazocinnolinones **V, VI** or imidazoquinazolinones **X, XI**, and also for discerning the structures **VII** and **IX**. The final proof of the formation of the imidazo-[1,5-*b*] cinnolinone system was obtained by XRD analysis of the single crystal of compound **VII** (see the figure).

The XRD investigation of molecule **VIIg** showed that the imidazopyridazine fragment is flat within 0.02 Å. The leveling of bond lengths N^{2}–C^{1} and N^{3}–C^{1} in the imidazole ring [1.335(3) and 1.333(3) Å, respectively, compared to the average value [11] for the C_{sp^2}=N bond 1.313 Å] makes it possible to represent the structure of the molecule as a superposition of a neutral and zwitter-ionic form **A**.



A

Evidently this results in the deep color of the substance. The amino group has a pyramidal configuration, the sum of the bond angles at the nitrogen atom equals 353°. The cyclohexenone fragment is present in the conformation unsymmetrical *semichair* (folding parameters [12] *S* 0.77, Θ 31.1°, Ψ 13.9°). The deviation of atoms C^{7} and C^{8} from the root-mean-square plane of the other ring atoms are –0.17 and 0.52 Å respectively. The close spatial location of the carbonyl group, aryl and phenyl substituents results in a significant steric repulsion between them [shortened intramolecular contacts C^{13}…C^{19} 3.27 Å (sum of van der Waals radii [13] 3.42 A), C^{18}···C^{19} 3.18 (3.42), C^{13}···C^{20} 3.13 (3.42), O^{1}···C^{19} 2.78, and O^{1}···C^{24} 2.89 Å]. Consequently the phenyl ring and C=O bond deviate from the bicycle plane [torsion angles $C^{13}C^{2}C^{3}C^{4}$ 7.0(5)°, $C^{4}C^{5}C^{6}O^{1}$ 6.5(4)°], the turn of the aromatic rings occurs [torsion angles $N^{3}C^{2}C^{13}C^{14}$ 54.7(3)°, $C^{5}C^{4}C^{19}C^{24}$ 67.5(3)°], and the deformation of the bond angles $N^{3}C^{2}C^{13}$ 120.1(2)°, $C^{3}C^{2}C^{13}$ 130.6(2)°, and the bond angles at the atom C^{4} [$C^{5}C^{4}C^{19}$ 125.9(2)° and $C^{3}C^{4}C^{19}$ 116.0(2)°]. On the other hand, between two π-systems of substituents stacking interaction might arise that might cause the certain flexion of the *para*-nitrophenyl substituent [torsion angle $C^{4}C^{19}C^{24}C^{23}$ 172.6(2)°].



Structure of compound **VIIg** according to XRD data.

In the crystal of molecule **VIIg** centrosymmetric dimers are formed owing to the intermolecular hydrogen bond N^{4}–H^{4Nb}···$N^{3'}$ [–*x*, 1 – *y*, 1 – *z*; H···N 2.09(4) Å, N–H···N 167(3)°] The dimers are linked with each other by a weaker intermolecular hydrogen bond N^{4}–H^{4Na}···$O^{2''}$ [*x*, 1.5 – *y*, 0.5 + *z*; H···O 2.34(3) Å, N–H···O 160(3)°].

The structure of compounds **V,VI, VIIg** indicates that the reactions of 1,2-diamino-4-phenylimidazole (**I**) with aromatic aldehydes **IIa–IIg** and dimedone (**III**) or 1,3-cyclohexanedione (**IV**) result in the formation of azoloazine system of only one type. In all studied processes the direction of the electrophilic attack corresponds to the interaction of the reaction site C^{5} in the diaminoazole molecule with the β-carbon atom, and of N^{1}-amino group with the carbonyl group of the enone formed by the condensation of the aldehyde with the cyclic diketone. Consequently the same cascade of processes occurs leading to the formation from diaminoimidazole, aldehyde, and the cyclic CH-acid of a pyridazine and not a pyrimidine ring in the composition of the fused system, as we formerly have found in the reactions of amine **I** with aromatic aldehydes and Meldrum's acid [6].

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer Specord M-82 from pellets with KBr. ^{1}H NMR spectra were registered on a spectrometer Varian Mercury VX-

200, solvent DMSO-d_6, internal reference TMS. Melting points were measured on a Koeffler heating block.

XRD analysis. Crystals of compound **VIIg** monoclinic, $C_{24}H_{21}N_5O_3$, at 20°C *a* 9.245(2), *b* 9.878(2), *c* 23.185(4) Å, β 98.93(2)°, *V* 2091.6(6) Å^3, M_r 427.46, *Z* 4, space group $P2_1/c$, d_{calc} 1.357 g/cm^3, μ(MoK_α) 0.093 mm^{-1}, *F*(000) 896. Parameters of the unit cell and the intensities of 16002 reflections (3669 independent, R_{int} 0.055) were measured on a diffractometer Xcalibur-3 (MoK_α radiation, CCD-detector, graphite monochromator, ω-scanning, $2\theta_{max}$ 50°). The structure was solved by the direct method using software SHELXTL [14]. The positions of hydrogen atoms were revealed from the difference synthesis of the electron density and refined in the *rider* model with $U_{iso} = nU_{eq}$ of the nonhydrogen atom linked to this hydrogen (n = 1.5 for the methyl group and 1.2 for the other hydrogen atoms). The hydrogen atoms involved in the hydrogen bonds were refined in the isotropic approximation. The structure was refined with respect to F^2 by the full-matrix least-squares method in the anisotropic approximation for nonhydrogen atoms till wR_2 0.157 for 3628 reflections (R_1 0.055 for 2064 reflections with $F > 4\sigma(F)$, *S* 0.940). Atomic coordinates and complete tables of bond distances and bond angles are deposited to the Cambridge Cristallographic Data Center (e-mail: deposit@ccdc.cam.ac.uk), CCDC 813671.

Reaction of 1,2-diamino-4-phenylimidazole (I) with benzaldehydes IIa–IId and dimedone (III). *a*. A mixture of 2 mmol of amine **I**, 2 mmol of an appropriate aldehyde **IIa–IId,** and 2 mmol of dimedone (**III**) in 5 ml of methanol was boiled for 2 h. The reaction mixture was cooled, compound **V** was filtered off.

b. A mixture of 2 mmol of amine **I**, 2 mmol of an appropriate aldehyde **IIa–IId,** and 2 mmol of dimedone (**III**) in 2 ml of DMF was boiled for 1 h. The reaction mixture was cooled, 5 ml of 2-propanol was added, compound **V** was filtered off and recrystallized from 2-propanol**.**

3-Amino-7,7-dimethyl-1,10-diphenyl-7,8-dihydroimidazo[1,5-*b*]cinnolin-9(5*H*,6*H*,10*H*)-one (Va). Yield 1.4 mmol (68%) (*a*), 1.1 mmol (53%) (*b*), mp 179–181°C (decomp.). IR spectrum, cm^{-1}: 3276–2660 (NH_2, CH_2), 1684 (CO). ^{1}H NMR spectrum, δ, ppm: 1.00, 1.03 s (3H, CH_3), 1.96–2.03 d.d (2H, CH_2, *J* 15.2, 16.6 Hz), 2.32 s (2H, CH_2), 5.06 s (1H, C^{10}H), 5.70 br.s (2H, NH_2), 7.01–7.56 m (10H, C_6H_5, C_6H_5). Mass spectrum: *m/z* (I_{rel}, %): 384.54 (30) $[M]^+$, 307 (17), 247 (20), 205 (10), 105 (77), 77 (100). Found, %: C 75.04; H 6.26; N 14.60. $C_{24}H_{24}N_4O$. Calculated, %: C 75.00; H 6.25; N 14.58.

3-Amino-7,7-dimethyl-1-phenyl-10-(4-methylphenyl)-7,8-dihydroimidazo[1,5-*b*]cinnolin-9(5*H*,6*H*,10*H*)-one (Vb). Yield 1.3 mmol (64%) (*a*), 1 mmol (51%) (*b*), mp 180–183°C (decomp.). IR spectrum, cm^{-1}: 3336–2660 (NH_2, CH_2), 1688 (CO). ^{1}H NMR spectrum, δ, ppm: 1.02, 1.03, 2.14 s (3H, CH_3), 1.96–2.01 d.d (2H, CH_2, *J* 12.8, 14.8 Hz), 2.22 s (2H, CH_2), 5.39 s (1H, C^{10}H), 5.66 br.s (2H, NH_2), 6.86–7.44 m (9H, C_6H_5, C_6H_4). Found, %: C 75.40; H 6.52; N 14.00. $C_{25}H_{26}N_4O$. Calculated, %: C 75.38; H 6.53; N 14.07.

3-Amino-7,7-dimethyl-1-phenyl-10-(4-methoxyphenyl)-7,8-dihydroimidazo[1,5-*b*]cinnolin-9(5*H*,6*H*,10*H*)-one (Vc). Yield 1.1 mmol (57%) (*a*), 0.8 mmol (47%) (*b*), mp 176–178°C (decomp.). IR spectrum, cm^{-1}: 3312–2660 (NH_2, CH_2), 1688 (CO). ^{1}H NMR spectrum, δ, ppm: 1.02, 1.03 s (3H, CH_3), 1.82–2.03 d.d (2H, CH_2, *J* 14.6, 15.8 Hz), 2.14 s (2H, CH_2), 3.61 s (3H, OCH_3), 5.33 s (1H, C^{10}H), 5.58 br.s (2H, NH_2), 6.71–7.41 m (9H, C_6H_5, C_6H_4). Found, %: C 72.47; H 6.28; N 13.50. $C_{25}H_{26}N_4O_2$. Calculated, %: C 72.46; H 6.28; N 13.53.

3-Amino-7,7-dimethyl-1-phenyl-10-(4-chlorophenyl)-7,8-dihydroimidazo[1,5-*b*]cinnolin-9(5*H*,6*H*,10*H*)-one (Ve). Yield 1.2 mmol (62%) (*a*), mp 193–195°C (decomp.). IR spectrum, cm^{-1}: 3280–2680 (NH_2, CH_2), 1684 (CO). ^{1}H NMR spectrum, δ, ppm: 1.02, 1.01 s (3H, CH_3), 1.76–2.03 d.d (2H, CH_2, *J* 14.8, 16.2 Hz), 2.13 s (2H, CH_2), 5.33 s (1H, C^{10}H), 5.66 br.s (2H, NH_2), 6.92–7.44 m (9H, C_6H_5, C_6H_4). Found, %: C 68.83; H 5.52; N 13.39. $C_{24}H_{23}ClN_4O$. Calculated, %: C 68.82; H 5.50; N 13.38.

3-Amino-7,7-dimethyl-1-phenyl-10-(4-fluorophenyl)-7,8-dihydroimidazo[1,5-*b*]cinnolin-9(5*H*,6*H*,10*H*)-one (Vf). Yield 1.3 mmol (65%) (*a*), mp 185–187°C (decomp.). IR spectrum, cm^{-1}: 3350–2860 (NH_2, CH_2), 1688 (CO). ^{1}H NMR spectrum, δ, ppm: 1.01 s (6H, CH_3), 1.75–2.03 d.d (2H, CH_2, *J* 13.6, 15.2 Hz), 2.13 s (2H, CH_2), 5.31 s (1H, C^{10}H), 5.64 br.s (2H, NH_2), 6.93–7.41 m (9H, C_6H_5, C_6H_4). Found, %: C 71.64; H 5.71; N 13.91. $C_{24}H_{23}FN_4O$. Calculated, %: C 71.64; H 5.72; N 13.93.

3-Amino-7,7-dimethyl-1-phenyl-10-(4-nitrophenyl)-7,8-dihydroimidazo[1,5-*b*]cinnolin-9-one (VIIg). Yield 1 mmol (48%) (*b*), mp 257–260°C (decomp.). IR spectrum, cm^{-1}: 3424–2896 (NH_2, CH_2), 1676 (CO), 1348 (NO_2). ^{1}H NMR spectrum, δ, ppm: 1.04 (6H, CH_3), 2.35 s (2H, CH_2), 2.82 s (2H, CH_2),

6.75–7.01 m (7H, C_6H_5, NH_2), 7.24–7.85 d.d (4H, C_6H_4, *J* 8.0, 8.0 Hz). Mass spectrum: *m/z* (I_{rel}, %): 427.52 (100) $[M]^+$, 381 (12), 140 (15), 77 (25). Found, %: C 67.15; H 5.37; N 16.35. $C_{24}H_{21}N_5O_3$. Calculated, %: C 67.15; H 5.36; N 16.32.

Reaction of 1,2-diamino-4-phenylimidazole (I) with benzaldehydes IIa, IIb, IId, IIe, IIg and 1,3-cyclohexanedione (IV). A mixture of 2 mmol of amine **I**, 2 mmol of an appropriate aldehyde **IIa, IIb, IId, IIe, IIg**, and 2 mmol of 1,3-cyclohexanedione (**IV**) in 5 ml of methanol was boiled for 2 h. The reaction mixture was cooled, compound **VI** was filtered off.

3-Amino-1,10-diphenyl-7,8-dihydroimidazo-[1,5-*b*]cinnolin-9(5*H*,6*H*,10*H*)-one (VIa). Yield 1.5 mmol (74%), mp 188–190°C (decomp.). IR spectrum, cm^{-1}: 3428–2844 (NH_2, CH_2), 1688 (CO). ^1H NMR spectrum, δ, ppm: 1.80 m (2H, CH_2), 2.36 m (4H, CH_2), 5.40 s (1H, C*10*H), 5.75 br.s (2H, NH_2), 7.00–7.56 m (10H, C_6H_5, C_6H_5). Mass spectrum: *m/z* (I_{rel}, %): 356.46 (100) $[M]^+$, 279 (15), 176 (10), 103 (12), 77 (10). Found, %: C 74.19; H 5.63; N 15.71. $C_{22}H_{20}N_4O$. Calculated, %: C 74.16; H 5.62; N 15.73.

3-Amino-1-phenyl-10-(4-methylphenyl)-7,8-dihydroimidazo[1,5-*b*]cinnolin-9(5*H*,6*H*,10*H*)-one (VIb). Yield 1.3 mmol (65%), mp 187–189°C (decomp.). IR spectrum, cm^{-1}: 3432–2872 (NH_2, CH_2), 1688 (CO). ^1H NMR spectrum, δ, ppm: 1.79 m (2H, CH_2), 2.15 s (3H, CH_3), 2.36 m (4H, CH_2), 5.35 s (1H, C*10*H), 5.71 br.s (2H, NH_2), 6.98–7.26 m (5H, C_6H_5), 7.20–7.55 d.d (4H, C_6H_4, *J* 7.4 Hz). Mass spectrum: *m/z* (I_{rel}, %): 370.49 (100) $[M]^+$, 293 (13), 260 (10), 196 (10), 155 (13), 119 (17), 91 (43), 77 (11). Found, %: C 74.62; H 5.95; N 15.15. $C_{23}H_{22}N_4O$. Calculated, %: C 74.60; H 5.95; N 15.14.

3-Amino-1-phenyl-10-(4-dimethylaminophenyl)-7,8-dihydroimidazo[1,5-*b*]cinnolin-9(5*H*,6*H*,10*H*)-one (VId). Yield 1.2 mmol (62%), mp 218–220°C (decomp.). IR spectrum, cm^{-1}: 3492–2796 (NH_2, CH_2), 1688 (CO). ^1H NMR spectrum, δ, ppm: 1.79 m (2H, CH_2), 2.36 m (4H, CH_2), 2.76 s (6H, CH_3), 5.36 s (1H, C*10*H), 5.68 br.s (2H, NH_2), 6.51–7.23 m (5H, C_6H_5), 6.93–7.57 d.d (4H, C_6H_4, *J* 7.6, 7.6 Hz). Found, %: C 72.23; H 6.75; N 15.59. $C_{24}H_{25}N_5O$. Calculated, %: C 72.18; H 6.27; N 17.54.

3-Amino-1-phenyl-10-(4-chlorophenyl)-7,8-dihydroimidazo[1,5-*b*]cinnolin-9(5*H*,6*H*,10*H*)-one (VIe). Yield 1.4 mmol (72%), mp 228–230°C (decomp.). IR spectrum, cm^{-1}: 3340–2872 (NH_2, CH_2), 1672 (CO). ^1H NMR spectrum, δ, ppm: 1.79 m (2H, CH_2), 2.36 m (4H, CH_2), 5.40 s (1H, C*10*H), 5.78 br.s (2H, NH_2), 7.10–7.50 d.d (4H, C_6H_4, *J* 7.8 Hz), 7.22 m (5H, C_6H_5). Mass spectrum: *m/z* (I_{rel}, %): 392.45 $[M+2]^+$ (27), 390.45 $[M]^+$ (80), 279 (100), 139 (15), 111 (18), 77 (24). Found, %: C 67.60; H 4.85; N 14.35. $C_{22}H_{19}ClN_4O$. Calculated, %: C 67.61; H 4.87; N 14.34.

3-Amino-1-phenyl-10-(4-nitrophenyl)-7,8-dihydroimidazo[1,5-*b*]cinnolin-9(5*H*,6*H*,10*H*)-one (VIg). Yield 1.2 mmol (60%), mp 208–210°C (decomp.). IR spectrum, cm^{-1}: 3340–2872 (NH_2, CH_2), 1676 (CO), 1344 (NO_2). ^1H NMR spectrum, δ, ppm: 1.79 m (2H, CH_2), 2.40 m (4H, CH_2), 5.53 s (1H, C*10*H), 5.91 br.s (2H, NH_2), 7.25 m (5H, C_6H_5), 8.04, 7.37 d.d (4H, C_6H_4, *J* 8.2, 8.2 Hz). Found, %: C 65.83; H 4.74; N 17.47. $C_{22}H_{19}N_5O_3$. Calculated, %: C 65.84; H 4.74; N 17.46.

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