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Reactions of 1,5-Diketones with 2-Aminobenzyl Alcohol and 2-Aminomethylaniline and Behavior of the Products in Oxidative Coupling

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Abstract—Reactions of some 1,5-diketones with 2-aminobenzyl alcohol and 2-aminomethylaniline follow double cyclization scheme with formation of pyridobenzoxazine and pyridoquinazoline derivatives, respectively. Oxidative coupling of the cyclization products with CH acids occurs at the 4-position of the pyridine ring.

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Reactions of 1,5-dicarbonyl compounds with such 1,2-binucleophiles as *o*-phenylenediamine, *o*-aminophenol, and 2-aminoethanol have been extensively studied. These reactions lead to the formation of double cyclization products belonging to the pyrido[1,2-*a*]benzimidazole and pyrido[3,2-*a*](benz)oxazole series [1, 2]. Successful double cyclization was also performed with some 1,3-binucleophiles, in particular with 3-aminopropan-1-ol and anthranilic acid [2], and derivatives of pyrido[2,1-*b*]oxazine and pyrido[1,2-*a*]-benzoxazin-6-one were obtained.

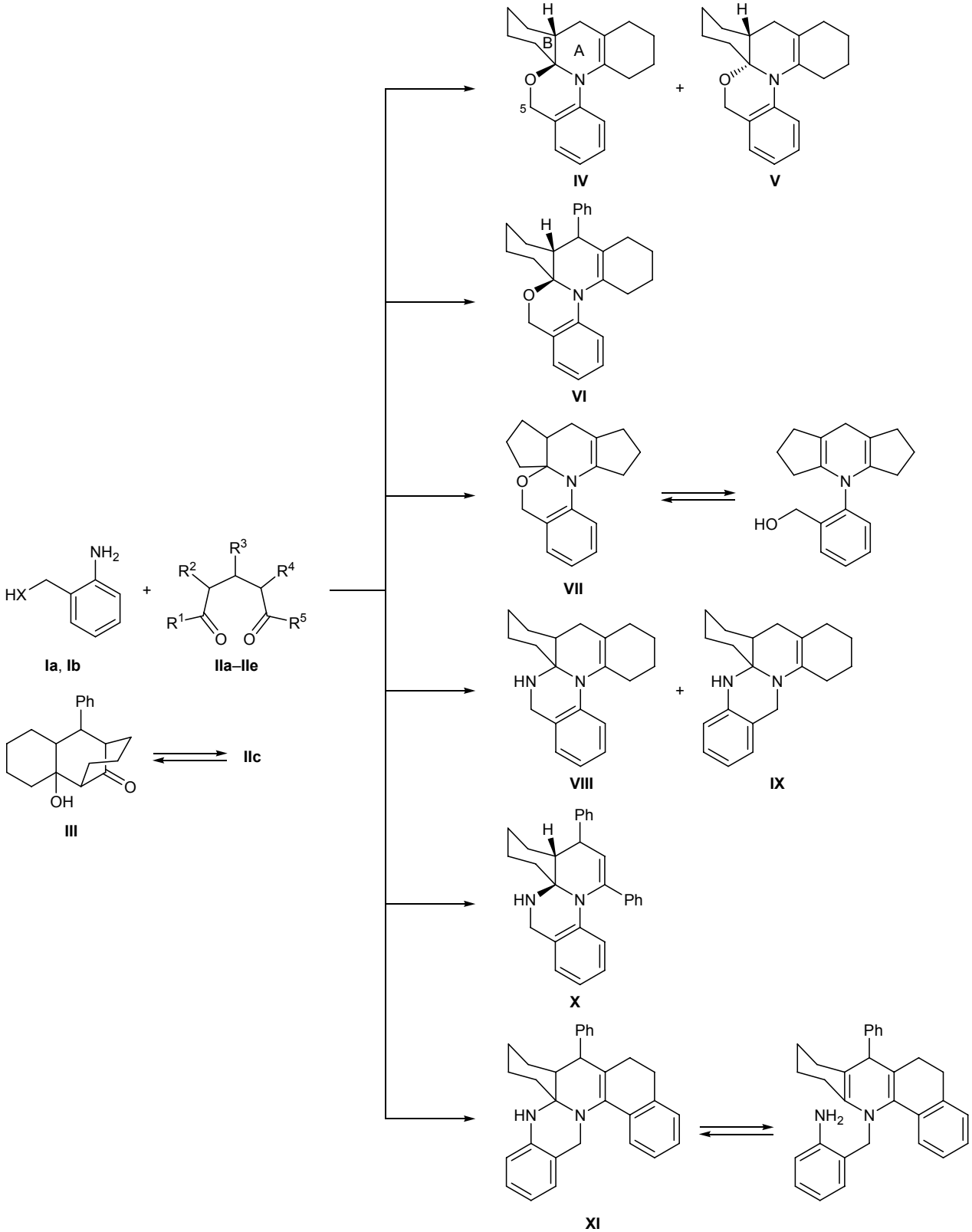
Reactions of such 1,3-binucleophiles as 2-aminobenzyl alcohol and 2-aminomethylaniline with 1,5-dicarbonyl compounds were not reported. It is well known that reactions of 2-aminobenzyl alcohol (**Ia**), 2-aminomethylaniline (**Ib**), and their analogs with monocarbonyl compounds generally involve cyclization to 1,3-benzoxazine [3, 4] and 1,3-benzodiazine structures [5, 6]. 1,2- and 1,3-Dicarbonyl compounds reacted with 2-aminobenzyl alcohol (**Ia**) at one carbonyl group, leading to diimines and enamines, respectively [7, 8]. Reactions of 2-aminomethylaniline (**Ib**) with 1,2-dicarbonyl compounds produced benzodiazepines or benzodiazines if only one carbonyl group was involved [9]. The cyclization with 1,3-dicarbonyl compounds also occurred at one carbonyl group with formation of 1,3-benzodiazines [10], whereas 1,4-dicarbonyl compounds gave rise to benzopyrimidine derivatives as a result of double cyclization [11]. We have found only one reported example of reaction of an analog of 2-aminomethylaniline (**Ib**) with a 1,5-dicarbonyl compound, 2-(2-oxoethyl)benzaldehyde, in which successive closure of two six-membered rings led to pyridobenzodiazine system [12].

In the present work we studied the behavior of some 1,5-diketones in reactions with compounds **Ia** and **Ib**. Taking into account the data of [1, 2], these reactions might be expected to follow double cyclization scheme with successive closure of hydrogenated pyridine and 1,3-oxazine (pyrimidine) rings.

As 1,5-dicarbonyl compounds we used diketones **IIa**–**IIe**; instead of diketone **IIc**, the reactions were carried out with more accessible product of its intramolecular aldolization–crotonization, hydroxy ketone **III**, which generated **IIc** via retro-aldol reaction. Compound **III** has already been used as synthetic equivalent of **IIc** in reactions with binucleophiles [13] (Scheme 1). The reactions of diketones **IIa**, **IIb**, **IId**, and **IIe** with 2-aminobenzyl alcohol (**Ia**) were carried out at 60–70°C, while compound **III** reacted with **Ia** in boiling xylene over a period of 3–4 days. Diketones **IIb**, **IId**, and **IIe** reacted with 2-aminomethylaniline (**Ib**) on heating in boiling benzene (15–16 h), whereas the reaction of **IIa** with **Ib** occurred at room temperature. In all cases, *p*-toluenesulfonic acid was added as catalyst.

All reactions followed the double cyclization scheme; however, they required a longer time and were characterized by lower selectivity and lower yields, as

Scheme 1.



I, X = O (**a**), NH (**b**); **II**, $R^1R^2 = R^4R^5 = (CH_2)_4$, R^3 = H (**a**); $R^1R^2 = R^4R^5 = (CH_2)_3$, R^3 = H (**b**); $R^1R^2 = R^4R^5 = (CH_2)_4$, R^3 = Ph (**c**); $R^1R^2 = (CH_2)_4$, $R^3 = R^5$ = Ph, R^4 = H (**d**); $R^1R^2 = (CH_2)_4$, R^3 = Ph, R^4R^5 = 1,2-(3,4-dihydronaphtho) (**e**).

compared to analogous reactions of the same diketones with *o*-aminophenol and *o*-phenylenediamine. Amino alcohol **Ia** reacted only with symmetric 1,5-diketones **IIa**–**IIc**, while fatty–aromatic diketones **IId** and **IIe** failed to react even under prolonged heating. Diamine **Ib** reacted only with diketones **IIa**, **IId**, and **IIe**. Nonequivalence of the primary amino groups in **Ib** and unsymmetrical structure of diketones **IId** and **IIe** gave rise to regioselectivity problem in both substrate and reagent. Prolonged heating of the reaction mixture of compounds **IIa** and **Ib** resulted in the formation of a mixture of cyclization products **VIII** and **IX** which could not be separated by chromatography (similar R_f values).

The IR spectra of **IV**–**VII** lacked absorption band typical of OH stretching vibrations, whereas compounds **VIII**–**XI** displayed an absorption band assignable to secondary amino group in support of their cyclic structure. Also, a single absorption band belonging to enamino group was observed in the region 1629–1677 cm^{-1} (the spectra of dihydropyridine structures with an opened ring contain two bands in that region).

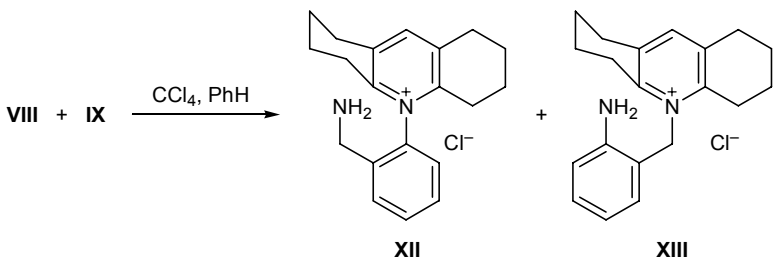
In the ^{1}H NMR spectra of **IV**–**VI** and **VIII**–**X** we observed signals from two nonequivalent benzylic protons (originating from compounds **Ia** and **Ib**) as two doublets in the region δ 4.16–4.87 ppm, ^2J = –12.6 to –19.0 Hz, which correspond to conformationally rigid benzoxazine (benzopyrimidine) structure. Compounds **IV** and **V** displayed in the ^{1}H NMR spectra two sets of signals; presumably, they were formed as mixtures of stereoisomers with *cis*- and *trans*-junction of the A and B rings (isomer ratio 2.7 : 1). According to the IR data, crystalline compounds **VII** and **XI** (KBr) have cyclic structure (closed oxazine and pyrimidine rings respectively). In going to solution in $CDCl_3$ for recording ^{1}H NMR spectra, opening of the heteroring occurs, and the corresponding benzylic protons become equivalent (a singlet is observed). Ready opening of partly hydrogenated oxazine ring in the double cyclization products obtained from diketones containing a five-membered ring was reported previously [1]; opening of imidazole ring was not observed under analogous conditions.

The ^{1}H NMR spectrum of mixture **VIII**/**IX** (2 : 3) contained four doublets belonging to nonequivalent benzylic protons, which was interpreted as formation of two isomers corresponding to different cyclization pathways. This assumption was confirmed by oxidation of mixture **VIII**/**IX** with carbon tetrachloride on heating in boiling benzene according to the procedure described in [14] (Scheme 2). In the ^{1}H NMR spectrum of pyridinium salt mixture **XII**/**XIII** (2 : 3) thus obtained we observed singlets from benzylic protons at δ 4.46 and 5.63 ppm. Comparison with the spectral data for *N*-benzyloctahydroacridinium salt [15] which displayed benzylic proton signal at δ 5.80 ppm (s) allowed us to assign the signal at δ 5.63 ppm to isomer **XIII**, and that at δ 4.46 ppm, to isomer **XII**.

In the ^{1}H NMR spectrum of **X**, the doublet signal from the aromatic 4-H proton is displaced appreciably upfield (δ 5.91 ppm). According to *ab initio* quantum-chemical calculations (6-31G* basis set, gas phase) for analogous double cyclization product of diketone **IId** with *o*-phenylenediamine, the 4-H proton is shielded due to magnetically anisotropic effect of the 6-substituent [16]. No such upfield shift was observed in the spectrum of **XI**; therefore, the latter was assigned the structure resulting from pyridine ring closure with participation of the benzylamino group; in this case, protons in the aromatic residue of diamine **Ib** are not shielded by fairly distant phenyl group.

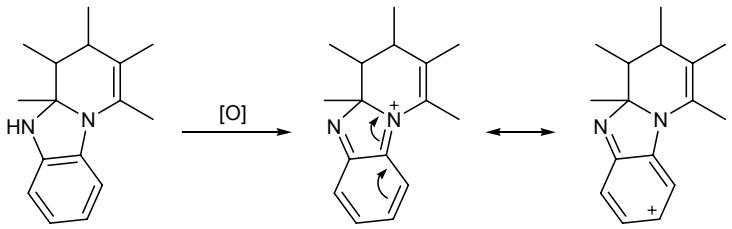
The structure of compound **VI** was unambiguously proved by X-ray analysis [17]. The formation of one isomer with *cis*-junction of the A and B rings is likely to be determined by additional stabilization effect produced by the phenyl substituent in position *3*. On the basis of these data we presumed *cis*-junction of the A and B rings in structurally related double cyclization products **X** and **XI**.

Scheme 2.



XII **XIII**

Scheme 3.



XIV **A**

We previously showed that double cyclization products like **XIV** (derived from 1,5-diketones and *o*-phenylenediamine) are capable of undergoing oxidation and oxidative coupling with a number of amines and CH acids; these reactions afforded heterocyclic *p*-quinone monoimines, *p*-quinone diimines and *p*-methylenequinone imines, respectively [18, 19]. A probable mechanism of oxidative coupling implied initial formation of pyridobenzimidazolium cation **A** stabilized by the donor vinylamino fragment (Scheme 3).

In order to extend the series of possible substrates for oxidative coupling and prove (or disprove) its mechanism proposed previously, we examined oxidative coupling of isomer mixture **IV**/**V** with malononitrile and indan-1,3-dione in ethanol in the presence of MnO_2 activated according to [20] (Scheme 4). The reaction was much slower (3 days at room temperature) and considerably less selective, and it followed a different pathway. We succeeded in isolating by column chromatography non-quinoid oxidative coupling products **XV** and **XVI** in a poor yield.

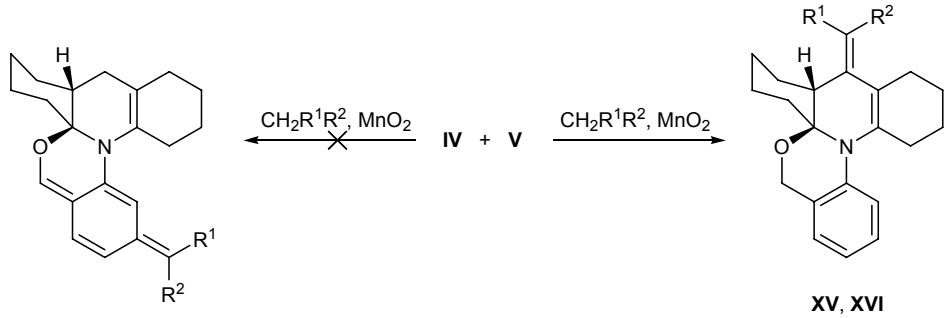
Taking into account published data on nucleophilic addition to pyridinium salts at the γ-position [21, 22], we presumed that the reaction involves reversible opening of the oxazine ring with formation of dihydropyridine structure **B**, its subsequent oxidation to pyridinium zwitterion **C**, and attack on the latter by CH acid anion at the γ-position to give nucleophilic addition product **D** which is readily oxidized to dihydropyridine **E** (Scheme 5). The final step is oxazine ring closure, and the substituent in the γ-position of the pyridine ring favors *cis*-junction of the A and B rings. However, we failed to properly confirm the proposed mechanism of oxidative coupling by carrying out the reaction of malononitrile with a simpler substrate, *N*-benzyldecahydroacridine, for the reaction was absolutely nonselective.

The IR spectrum of **XV** contained an absorption band at 2207 cm^{-1} due to stretching vibrations of conjugated cyano group, and compound **XVI** displayed an absorption band at 1717 cm^{-1} due to conjugated carbonyl group in the five-membered ring. Enamine absorption band had low intensity and was displaced toward lower frequencies, as compared to the initial compounds, as a result of extension of the conjugation chain. The IR spectra of both compounds **XV** and **XVI** lacked OH absorption. In the ^1H NMR spectra of **XV** and **XVI** benzylic protons in the oxazine ring resonated as doublets with the corresponding geminal coupling constant, whereas no signals assignable to quinoid protons were present; therefore, quinoid structure was unambiguously ruled out.

EXPERIMENTAL

The IR spectra were recorded in KBr on a Spectrum BX-II spectrometer. The ^1H NMR spectra were measured on a Bruker AC-250 spectrometer operating at

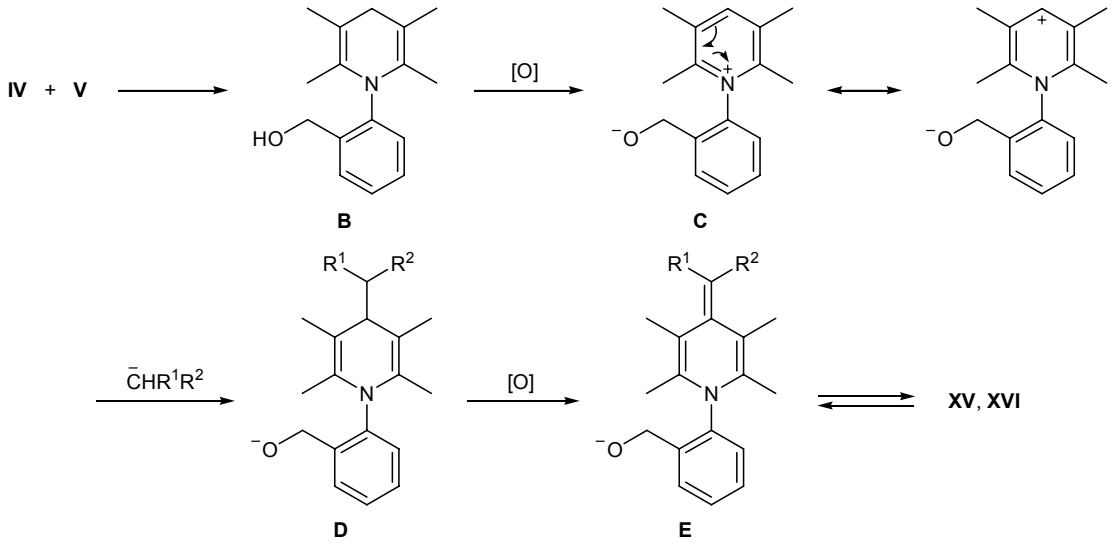
Scheme 4.



XV, **XVI**

XV, $R^1 = R^2 = CN$; **XVI**, R^1R^2 = phthaloyl.

Scheme 5.



250 MHz; tetramethylsilane was used as internal reference, and $CDCl_3$, as solvent. The elemental compositions were determined on a Flash EA 1112 CHN/MAS200 CHN analyzer. HPLC analyses were run on an HP 1100 LC/MSD instrument equipped with a 4×125-mm Hypersil ODS column and a diode array detector (eluent propan-2-ol–water, 50:50, flow rate 0.3 ml/min; temperature 55°C; API-ES ion source, positive polarity). The melting points were determined in capillaries on a Boetius melting point apparatus. The progress of reactions and the purity of products were monitored by TLC on Silufol UV-254 and Sorbfil plates using petroleum ether–ethyl acetate as eluent. The products were isolated and purified by column chromatography and preparative thin-layer chromatography (25×30-cm plates coated with aluminum oxide of activity grade II according to Brockmann; layer thickness 1.5 mm, sample weight 0.25 g).

All compounds were synthesized following a general procedure; only their isolation and purification procedures were different. Commercially available reagents (Aldrich, Fluka) were used without additional purification.

Compounds IV–XI (*general procedure*). Compound **IIa**, **IIb**, **IId**, or **IIe**, 1.0 mmol, was dissolved in 15 ml of benzene (compound **IIc** was dissolved in 15 ml of *p*-xylene), 1.1 mmol of amine **Ia** or **Ib** and 0.01 mmol of *p*-toluenesulfonic acid were added, and the mixture was kept for 150 h at room temperature (**VII**–**IX**), heated for 90 h at 60°C on a water bath (**IV**, **V**), or heated under reflux for 15 h (**X**, **XI**) or 35 h in a flask equipped with a Dean–Stark trap (**VI**) until the initial compound disappeared (TLC). The solvent was distilled off under reduced pressure, and the residue was subjected to column chromatography using petroleum ether as eluent (**IV**–**IX**); in the syntheses of **X** and **XI**, the residue was treated with 5 ml of ethanol, and almost pure product separated in 10 h and was recrystallized from ethanol. Compounds **IV**, **V**, **VIII**, and **IX** were isolated as oily substances, and the others were crystalline substances.

7,8,9,10,10a,11,12,13,14,15-Decahydro-5*H*-[3,1]-benzoxazino[2,1-*e*]acridine [(6a*S*,10a*S*)-IV, *cis*-A,B; (6a*R*,10a*S*)-V, *trans*-A,B]. Yield 32 %. IR spectrum: ν 1676 cm^{-1} (C^{11a}=C^{15a}). ^1H NMR spectrum, δ, ppm: 1.00–2.30 m (19H), 4.52 d (1H, 5-H_{ax} in **IV**, J = –13.2 Hz), 4.58 d (1H, 5-H_{eq} in **IV**, J = –13.2 Hz), 4.71 d (1H, 5-H_{ax} in **V**, J = –13.5 Hz), 4.87 d (1H, 5-H_{eq} in **V**, J = –13.5 Hz), 6.74 d (1H, 1-H, J = 8.0 Hz), 6.87 t (1H, 3-H, J = 8.0 Hz), 7.02 d (1H, 4-H, J = 8.0 Hz), 7.13 t (1H, 2-H, J = 8.0 Hz). Found, %: C 79.95; H 8.16; N 5.08. *m/z* 296 $[M + H]^+$. $C_{20}H_{25}NO$. Calculated, %: C 81.31; H 8.53; N 4.74. *M* 295.42.

11-Phenyl-7,8,9,10,10a,11,12,13,14,15-decahydro-5*H*-[3,1]benzoxazino[2,1-*e*]acridine (VI). Yield 41%, mp 160–162°C. IR spectrum: ν 1678 cm^{-1} (C^{11a}=C^{15a}). ^1H NMR spectrum, δ, ppm: 1.50–2.00 m (16H), 2.65 d (1H, 15-H_{eq}, J = 16.0 Hz), 3.04 d (1H, 11-H, J = 9.0 Hz), 4.40 d (1H, 5-H_{ax}, J = –12.6 Hz), 4.55 d (1H, 5-H_{eq}, J = –12.6 Hz), 6.80 t (2H, 7-H, 8-H, J = 8.0 Hz), 7.00–7.24 m (7H, H_{arom}). Found, %: C 83.92; H 7.69; N 3.70. *m/z* 372 $[M + H]^+$. $C_{26}H_{29}NO$. Calculated, %: C 84.06; H 7.87; N 4.31. *M* 371.51.

7,8,9,9a,10,11,12,13-Octahydro-5*H*-dicyclopenta-[2,3:5,6]pyrido[1,2-*a*][3,1]benzoxazine (VII). Yield

44%, mp 86–88°C. IR spectrum: ν 1678 cm^{-1} (C^{10a}=C^{13a}). ^1H NMR spectrum, δ, ppm: 1.59–2.79 m (14H), 4.92 br.s (2H, 5-H), 6.91–7.16 m (4H, H_{arom}). Found, %: C 80.98; H 8.23; N 5.20. *m*/*z* 268 $[M + H]^+$. $C_{18}H_{21}NO$. Calculated, %: C 80.86; H 7.92; N 5.24. *M* 267.37.

5,6,7,8,9,10,10a,11,12,13,14,15-Dodecahydroquinazolino[2,1-*e*]acridine (VIII) and 5,6,7,8,9,9a,-10,11,12,13,14,16-dodecahydroquinazolino[2,3-*e*]-acridine (IX). Yield 31%. IR spectrum, ν, cm^{-1}: 3415 (NH), 1668 (C=C). ^1H NMR spectrum, δ, ppm: 1.25–2.50 m (19H), 4.16 d (1H, 5-H_{ax} in **VIII**, *J* = –17.3 Hz), 4.26 d (1H, 5-H_{eq} in **VIII**, *J* = –17.3 Hz), 4.23 d (1H, 16-H_{ax} in **IX**, *J* = –19.0 Hz), 4.31 d (1H, 16-H_{eq} in **IX**, *J* = –19.0 Hz), 6.59 m (2H, 2-H, 3-H), 6.85 d (1H, 1-H, *J* = 7.2 Hz), 7.00 d (1H, 4-H, *J* = 7.3 Hz). Found, %: C 81.85; H 9.28; N 9.32. *m*/*z* 295 $[M + H]^+$. $C_{20}H_{26}N_2$. Calculated, %: C 81.59; H 8.90; N 9.51. *M* 294.43.

6,8-Diphenyl-8,8a,9,10,11,12,13,14-octahydroquinolino[1,8a-*a*]quinazoline (X). Yield 34%, mp 175–176°C. IR spectrum, ν, cm^{-1}: 3404 (NH), 1646 (C^6=C^7). ^1H NMR spectrum, δ, ppm: 1.20–2.35 m (9H), 3.33 d.d (1H, 8-H, *J* = 9.6, 5.5 Hz), 3.41 s (1H, NH), 4.00 d (1H, 14-H_{ax}, *J* = –16.8 Hz), 4.16 d (1H, 14-H_{eq}, *J* = –16.8 Hz), 4.73 d (1H, 7-H, *J* = 5.5 Hz), 5.91 d (1H, 4-H, *J* = 7.8 Hz), 6.51 t (1H, 3-H, *J* = 7.7 Hz), 6.58 d (1H, 1-H, *J* = 7.3 Hz), 6.93 t (1H, 2-H, *J* = 7.4 Hz), 7.15–7.40 m (10H, H_{arom}). Found, %: C 85.50; H 7.06; N 7.25. *m*/*z* 392 $[M + H]^+$. $C_{28}H_{28}N_2$. Calculated, %: C 85.67; H 7.19; N 7.14. *M* 392.54.

10-Phenyl-5,6,7,8,9,9a,10,11,12,18-decahydrobenzo[*c*]quinazolino[3,2-*f*]acridine (XI). Yield 55%, mp 162–164°C. IR spectrum, ν, cm^{-1}: 3430 (NH), 1630 (C^{10a}=C^{16b}). ^1H NMR spectrum, δ, ppm: 1.2–2.64 m (13H), 3.14 s (1H, 10-H), 4.37 br.s (2H, 18-H), 6.52 t (1H, 2-H, *J* = 7.6 Hz), 6.76 d (1H, 4-H, *J* = 7.6 Hz), 6.93 t (1H, 3-H, *J* = 7.6 Hz), 7.14–7.36 m (9H, H_{arom}), 7.46 d (1H, 1-H, *J* = 7.6 Hz). Found, %: C 86.34; H 7.42; N 6.55. *m*/*z* 419 $[M + H]^+$. $C_{30}H_{30}N_2$. Calculated, %: C 86.08; H 7.22; N 6.69. *M* 418.57.

10-[2-(Aminomethyl)phenyl]-1,2,3,4,5,6,7,8-octahydroacridinium chloride (XII) and 10-(2-aminobenzyl)-1,2,3,4,5,6,7,8-octahydroacridinium chloride (XIII). Mixture **VIII**/**IX**, 1.0 mmol, was dissolved in 10 ml of benzene, 4.0 mmol of carbon tetrachloride was added, and the mixture was heated for 3 h under reflux until the initial compounds disappeared (TLC). The solvent was distilled off under reduced pressure, and the crystalline residue was a mixture of acridinium chlorides **XII** and **XIII**. Yield 89%. ^1H NMR spectrum, δ, ppm: 1.25–3.20 m (16H), 3.63 d (1H, 4-H_{eq} in **XII**, *J* = –14.2 Hz), 4.46 s (2H, C$H_2$$NH_2$ in **XII**), 5.63 s (2H, CH_2N in **XIII**), 6.50–7.78 m (H_{arom}), 7.83 s (1H, 9-H in **XIII**), 7.94 s (1H, 9-H in **XII**).

Compounds XV and XVI. Stereoisomer mixture **IV**/**V**, 1.0 mmol, was dissolved in 8 ml of ethanol, 1.2 mmol of malononitrile or indan-1,3-dione was added, the mixture was stirred until it became homogeneous, 15 mmol of activated MnO_2 was added, and the mixture was kept at room temperature until the initial compound disappeared (TLC; 72 h in the synthesis of **XV** or 120 h in the synthesis of **XVI**). The precipitate of MnO_2 was filtered off and washed with ethanol until colorless washings. The filtrate was combined with the washings and evaporated, the residue was diluted with water, and the precipitate was filtered off, washed with water (3×5 ml), dried, and subjected to column chromatography on aluminum oxide using petroleum ether–ethyl acetate (2:1) as eluent. A yellow fraction was collected and evaporated to isolate compound **XV** or **XVI** as yellow crystals.

8,9,10,10a,12,13,14,15-Octahydro-5*H*-[3,1]benzoxazino[2,1-*e*]acridin-11(7*H*)-ylidenemalononitrile (XV). Yield 23%, mp 165–167°C. IR spectrum, ν, cm^{-1}: 2207 (CN), 1605 (C^{11a}=C^{15a}). ^1H NMR spectrum, δ, ppm: 1.20–2.50 m (17H), 4.77 d (1H, 5-H_{ax}, *J* = –12.3 Hz), 4.89 d (1H, 5-H_{eq}, *J* = –12.3 Hz), 6.97 d (1H, 1-H, *J* = 8.0 Hz), 7.13 t (1H, 3-H, *J* = 8.0 Hz), 7.18 d (1H, 4-H, *J* = 8.0 Hz), 7.22 t (1H, 2-H, *J* = 8.0 Hz). Found, %: C 77.39; H 6.24; N 11.85. *m*/*z* 358 $[M + H]^+$. $C_{23}H_{23}N_3O$. Calculated, %: C 77.28; H 6.49; N 11.76. *M* 357.45.

2-{8,9,10,10a,12,13,14,15-Octahydro-5*H*-[3,1]-benzoxazino[2,1-*e*]acridin-11(7*H*)-ylidene}-1*H*-indene-1,3(2*H*)-dione (XVI). Yield 18%, mp 100–102°C. IR spectrum, ν, cm^{-1}: 1717 (C=O), 1612 (C^{11a}=C^{15a}). ^1H NMR spectrum, δ, ppm: 1.25–2.10 m (17H), 4.73 d (1H, 5-H_{ax}, *J* = –12.5 Hz), 4.76 d (1H, 5-H_{eq}, *J* = –12.5 Hz), 6.92 d (1H, 1-H, *J* = 8.0 Hz), 7.06 t (1H, 3-H, *J* = 8.0 Hz), 7.12 d (1H, 4-H, *J* = 8.0 Hz), 7.17 t (1H, 2-H, *J* = 8.0 Hz), 7.50–7.73 m (4H, H_{arom}). Found, %: C 79.45; H 6.39; N 2.96. *m*/*z* 438 $[M + H]^+$. $C_{29}H_{27}NO_3$. Calculated, %: C 79.61; H 6.22; N 3.20. *M* 437.53.

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