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Synthesis of Benzo[*f*]quinoline Derivatives by Condensation of *N*-Arylmethylene-2-naphthylamines with Acetylcyclohexane and 1-Acetylcyclohexene

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Abstract—*N*-Arylmethylene-2-naphthylamines react with acetylcyclohexane and 1-acetylcyclohexene under mild conditions to afford 2-aryl-2-(2-naphthylamino)ethyl cyclohexyl and 1-cyclohexenyl ketones, respectively. Under more severe conditions (110°C), the reaction is accompanied by cyclization with formation of 3-aryl-1-cyclohexyl(or 1-cyclohexenyl)benzo[*f*]quinolines. Proper choice of the amount of catalyst, temperature, and reaction time allows isolation of intermediate 3-aryl-1-cyclohexyl(or 1-cyclohexenyl)-3,4-dihydrobenzo[*f*]quinolines.

Quinoline derivatives are structural analogs of alkaloids [1], enzyme inhibitors [2], and antibiotics [3]; therefore, they should exhibit versatile biological activity. In addition, these compounds are good antioxidants [4] and antirads [5] for polymeric materials. Interest in quinoline derivatives and analogs continuously increases. Some benzoquinolines were found to possess good complexing [6] and fluorescent properties [7].

A known procedure for the synthesis of benzo[*f*]quinolines is based on the condensation of Schiff bases derived from 2-naphthylamine with various aliphatic, aromatic, and heterocyclic methyl ketones [8–12]. It was found that the formation of benzo[*f*]quinolines is a multistep process [12]. The reaction of *N*-arylmethylene-2-naphthylamines with methyl ketones of the cycloaliphatic series has been studied poorly. We previously synthesized benzo[*f*]quinoline derivatives by condensation of *N*-arylmethylene-2-naphthylamines with acetylcyclohexane and 1-acetylcyclohexene [13, 14]. In doing so, either no intermediate products were isolated at all or they were isolated in very small amounts. In the present work we performed condensations of acetylcyclohexane (**I**) and 1-acetylcyclohexene (**II**) with Schiff bases **IIIa**–**IIIh** of the 2-naphthylamine series using larger amounts of the initial reactants and tried to isolate and examine intermediate products by varying the reaction conditions.

Condensations of Schiff bases with ketones are catalyzed by acids. Proton addition to a Schiff base yields the corresponding cation in which the positive charge is delocalized over the azomethine nitrogen and carbon atoms. Therefore, such cations readily take up even weakly nucleophilic ketones. The reaction mechanism is analogous to the acid-catalyzed aldol addition pattern. The reaction involves the methyl group in methyl ketone, so that the latter acts as CH acid. Amino ketones **IVa**–**IVf** and **Va**–**Vg** thus formed are the first intermediate products in the synthesis of benzo[*f*]quinolines (see scheme). The double bond in 1-acetylcyclohexene is conjugated with the carbonyl group; as a result, polarization of the C=O bond increases, and protons in the acetyl group become more labile. Therefore, the yields of amino ketones from 1-acetylcyclohexene are greater, and the condensation occurs at a lower temperature, as compared to acetylcyclohexane (Table 1). Amino ketones **IVa**–**IVf** and **Va**–**Vg** were obtained by heating the reactants in ethanol at 40–60°C in the presence of a catalytic amount of hydrochloric acid.

When the condensation was carried out under more severe conditions, i.e., by heating equimolar amounts of the reactants in toluene at 110°C in the presence of hydrochloric acid as catalyst, the products were the corresponding benzo[*f*]quinolines **VIa**–**VIh** and **VIIa**–**VIIh**. The cyclization was accompanied by formation of tarry products. Benzo[*f*]quinolines **VI** and **VII** can also be obtained from amino ketones **IV** and **V** under the same conditions; in this case, tarring occurred to a lesser extent. The cyclization of amino ketones **IV** and **V** to benzo[*f*]quinoline derivatives **VI** and **VII** involves electrophilic attack by the carbonyl carbon atom on the naphthalene α-carbon atom, followed by dehydration and dehydrogenation. It should be noted that the condensation of Schiff bases

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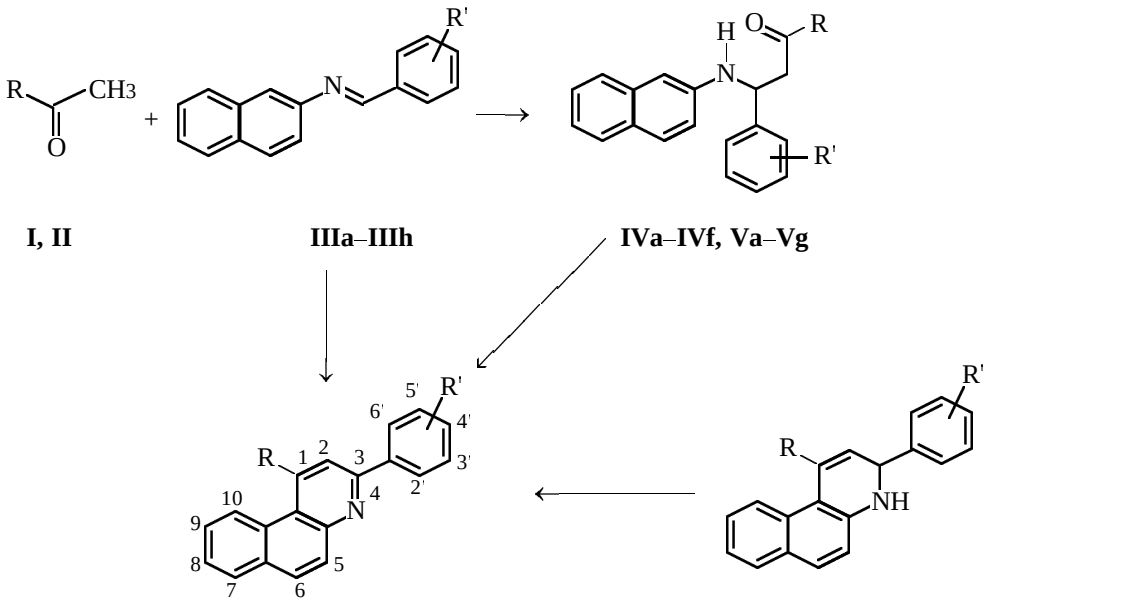

I, II **IIIa–IIIh** **IVa–IVf, Va–Vg**

VIa–VIh, VIIa–VIIh **VIIIb, VIIIc, VIIIe, VIIIf, IXc, IXe, IXf**

I, IV, VI, VIII, R = cyclohexyl; **II, V, VII, IX**, R = 1-cyclohexenyl; **III–IX**, R' = H (**a**), 4-F (**b**), 4-Cl (**c**), 4-Br (**d**), 4-NO_2 (**e**), 3-NO_2 (**f**), 4-OH (**g**), 4-NH_2 (**h**).

with acetophenone requires milder conditions (heating on a water bath for 10–15 min) and is not accompanied by tarring. Presumably, the cyclohexane and cyclohexene rings in methyl ketones **I** and **II** create stronger steric hindrances to cyclization, as compared to the benzene ring in acetophenone.

By varying the reaction conditions in the condensation of ketones **I** and **II** with *N*-arylmethylene-2-naphthylamines, namely the amount of the catalyst, temperature, and reaction time, we succeeded in isolating dihydro derivatives **VIIIb, VIIIc, VIIIe, VIIIf, IXc, IXe**, and **IXf** which are also intermediate products in the synthesis of benzo[*f*]quinolines **VI** and **VII**. Compounds **VIII** and **IX** were obtained by heating a mixture of the corresponding Schiff base and ketone in boiling ethanol over a period of 3–6 h or amino ketone over a period of 2–3 h.

The IR spectra of amino ketones **IVa**–**IVf** and **Va**–**Vg** contain absorption bands in the region 1680–1660 cm^{-1} due to carbonyl stretching vibrations; absorption in the region 3400–3390 cm^{-1} corresponds to stretching vibrations of the N–H group; stretching vibrations of the aromatic C–H bonds give rise to absorption bands at 3090–3000 cm^{-1}; and strong absorption bands from aromatic C=C bonds appear at 1630–1500 cm^{-1}. The IR spectra of dihydro derivatives **VIIIb**, **VIIIc**, **VIIIe**, **VIIIf**, **IXc**, **IXe**, and **IXf** lack absorption in the region 1680–1660 cm^{-1}, in keeping with their cyclic structure. A set of very strong absorption bands in the spectra of **VIII** and **IX** originates from out-of-plane bending vibrations of the aromatic C–H bonds. The region 1500–900 cm^{-1} contains a number of closely located bands due to vibrations of aromatic C–H bonds and stretching vibrations of the C–N bond. In the IR spectra of benzo[*f*]quinolines **VI** and **VII** we observed no absorption bands at 1680 and 3400 cm^{-1}, which are typical of C=O and N–H bonds, respectively. In the region corresponding to C=C stretching vibrations, strong triplet bands at 1580–1550 cm^{-1} and a strong band at 1490–1480 cm^{-1} were present. Absorption bands at 2880–2830 cm^{-1} belong to stretching vibrations of aliphatic C–H bonds in the cyclohexane (cyclohexene) rings. A weak band at 1630 cm^{-1} in the spectra of **VIIa**–**VIIh** was assigned to vibrations of the C=C bond in the cyclohexene ring.

The UV spectra of amino ketones **IV** and **V** (Table 2) consist of three bands and resemble the spectrum of 2-naphthylamine [λ_{max}, nm (log ε): 246 (4.38), 280 (3.66), 342 (3.28)]. Taking into account the above similarity and high absorption intensity, all these bands may be attributed to $\pi\rightarrow\pi^*$ transitions in the benzene rings. Weak bands due to $n\rightarrow\pi^*$ transition involving lone electron pair on the nitrogen atom are overlapped by the strong $\pi\rightarrow\pi^*$-transition bands. Substituents in the benzene ring almost do not affect the shape of the spectral curve: only a slight shift of the absorption maxima is observed. The UV spectra

Table 1. Yields, melting points, and elemental analyses of compounds **IV**–**IX**

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
IVa	67	165–166	84.02	7.48	4.08	$C_{25}H_{27}NO_2$	84.00	7.60	3.91
IVb	59	176–177	79.98	7.00	3.72	$C_{25}H_{26}FNO$	80.01	6.93	3.73
IVc	60	181–182	76.51	6.89	3.50	$C_{25}H_{26}ClNO$	76.50	6.68	3.58
IVd	58	187–188	68.80	6.04	3.16	$C_{25}H_{26}BrNO$	68.79	5.99	3.21
IVe	49	170–171	74.70	6.58	6.91	$C_{25}H_{26}N_2O_3$	74.61	6.50	6.96
IVf	43	175–176	74.58	6.52	6.99	$C_{25}H_{26}N_2O_3$	74.61	6.50	6.96
Va	72	164–165	84.60	7.02	3.97	$C_{25}H_{25}NO$	84.52	7.04	3.94
Vb	65	173–174	80.50	6.50	3.71	$C_{25}H_{24}FNO$	80.44	6.43	3.75
Vc	67	182–183	77.08	6.18	3.62	$C_{25}H_{24}ClNO$	77.00	6.19	3.59
Vd	63	192–193	69.11	5,63	3.25	$C_{25}H_{24}BrNO$	69.13	5.56	3.22
Ve	60	168–169	74.99	6.21	7.05	$C_{25}H_{24}N_2O_3$	75.01	5.99	7.00
Vf	49	171–172	74.97	6.07	6.98	$C_{25}H_{24}N_2O_3$	75.01	5.99	7.00
Vg	61	162–163	80.82	6.75	3.72	$C_{25}H_{25}NO_2$	80.87	6.73	3.77
VIa	72	140–141	88.94	6.90	4.02	$C_{25}H_{23}N$	88.98	6.86	4.10
VIb	68	166–167	84.54	6.18	3.99	$C_{25}H_{22}FN$	84.52	6.20	3.94
VIc	64	169–170	80.80	5.95	3.79	$C_{25}H_{22}ClN$	80.73	5.96	3.76
VId	54	181–182	72.11	5.27	3.92	$C_{25}H_{22}BrN$	72.10	5.32	3.96
VIe	49	174–175	78.57	5.83	7.38	$C_{25}H_{22}N_2O_2$	78.51	5.79	7.32
VIf	46	172–173	78.57	5.74	7.38	$C_{25}H_{22}N_2O_2$	78.51	5.79	7.32
VIg	60	169–170	84.92	6.59	3.99	$C_{25}H_{23}NO$	84.96	6.66	3.96
VIh	63	175–176	85.14	6.91	7.97	$C_{25}H_{24}N_2$	85.10	6.86	7.95
VIIa	64	138–139	89.63	6.29	4.05	$C_{25}H_{21}N$	89.55	6.27	4.18
VIIb	59	169–170	84.93	5.70	4.01	$C_{25}H_{20}FN$	84.99	5.66	3.96
VIIc	61	173–174	81.15	5.45	3.83	$C_{25}H_{20}ClN$	81.17	5.44	3.79
VIId	63	182–183	72.51	4.85	3.32	$C_{25}H_{20}BrN$	72.47	4.86	3.38
VIIe	38	178–179	78.89	5.33	7.36	$C_{25}H_{20}N_2O_2$	78.93	5.29	7.36
VIIf	42	175–176	78.87	5.28	7.39	$C_{25}H_{20}N_2O_2$	78.93	5.29	7.36
VIIg	60	168–169	85.51	6.06	3.98	$C_{25}H_{21}NO$	85.48	5.98	3.99
VIIh	63	172–173	85.74	6.32	8.02	$C_{25}H_{22}N_2$	85.72	6.28	8.00
VIIIb	28	217–218	84.08	6.78	3.89	$C_{25}H_{24}FN$	84.04	6.72	3.92
VIIIc	22	211–212	80.29	6.45	3.80	$C_{25}H_{24}ClN$	80.33	6.42	3.75
VIIId	36	215–216	78.17	6.31	7.30	$C_{25}H_{24}N_2O_2$	78.10	6.29	7.29
VIIIe	33	203–204	78.14	6.31	7.33	$C_{25}H_{24}N_2O_2$	78.10	6.29	7.29
IXc	29	210–211	80.80	5.99	3.75	$C_{25}H_{22}ClN$	80.77	5.92	3.77
IXe	23	218–219	78.47	5.86	7.28	$C_{25}H_{22}N_2O_2$	78.52	5.79	7.32
IXf	25	205–206	78.58	5.84	7.27	$C_{25}H_{22}N_2O_2$	78.52	5.79	7.32

of dihydro derivatives **VIII** and **IX** are also characterized by the presence of three strong absorption bands belonging to $\pi\rightarrow\pi^*$-electronic transitions in the naphthalene core, but their maxima are appreciably displaced to longer wavelength relative to the corresponding maxima of amino ketones **IV** and **V**. Presumably, the $\pi\rightarrow\pi^*$ transitions in molecules **VIII** and **IX** are contributed by lone electron pair on the nitrogen atom [15]. Substituents in the benzene ring weakly affect the position of the main absorption bands. The UV spectra of benzo[*f*]quinolines **VI** and **VII** contain bands typical of isoelectronic phenanthrene [16]. Replacement of the CH group in phenanthrene by nitrogen atom leads to increase in the intensity and red shift of the absorption maxima due to inductive effect of the nitrogen atom and reduction in the molecular symmetry. The absorption curves of benzo[*f*]quinoline derivatives **VI** and **VII** are systems including three bands: α, *p*, and β; the β-band is the most intense, and the α-band is characterized by a distinct vibronic structure. The absorption maxima in the UV spectra of cyclohexenyl-

Table 2. UV spectra of compounds **IVa**–**IVd, Va, Vc**–**Vf, VIa, VIc**–**VIf, VIIa, VIIc**–**VIIf, VIIIb, VIIIc, IXc**, and **IXd**

Comp. no.	λ_{max}, nm (log ε)	Comp. no.	λ_{max}, nm (log ε)
IVa	242 (4.53), 280 (3.87), 344 (3.46)	**VIe**	285 (4.58), 336 (3.79), 367 (3.64)
IVb	244 (4.41), 282 (3.94), 346 (3.31)	**VIf**	280 (4.46), 332 (3.53), 360 (3.59)
IVc	243 (4.62), 282 (3.96), 346 (3.43)	**VIIa**	281 (4.70), 336 (3.70), 362 (3.72)
IVd	244 (4.59), 284 (3.66), 345 (3.38)	**VIIc**	282 (4.62), 346 (3.61), 364 (3.64)
Va	243 (4.61), 284 (3.97), 348 (3.42)	**VIId**	283 (4.57), 348 (3.62), 365 (3.58)
Vc	240 (4.65), 284 (3.89), 349 (3.40)	**VIIe**	292 (4.48), 345 (4.26), 370 (4.03)
Vd	247 (4.58), 286 (3.92), 350 (3.39)	**VIIf**	284 (4.60), 347 (3.86), 363 (3.78)
Ve	248 (4.70), 288 (3.79), 352 (3.34)	**VIIIb**	258 (4.68), 309 (3.78), 373 (3.48)
Vf	246 (4.65), 283 (3.85), 347 (3.38)	**VIIIc**	259 (4.63), 308 (3.69), 376 (3.52)
VIa	278 (4.63), 330 (3.65), 359 (3.61)	**IXc**	266 (4.67), 316 (3.79), 380 (3.60)
VIc	280 (4.62), 334 (3.59), 360 (3.67)	**IXd**	268 (4.62), 318 (3.61), 372 (3.58)
VId	281 (4.42), 334 (3.63), 361 (3.60)		

Table 3. ^{1}H NMR spectra of benzo[*f*]quinoline derivatives **VIa**–**VIf** and **VIIa**–**VIIh**, δ, ppm (*J*, Hz)

Comp. no.	Benzoquinoline system				$R'C_6H_4$		R			
	H^2, s	H_{5-7}, m	$H^{8,9}$, d	H^{10}, d	$H^{2'}$, $H^{6'}$, m	$H^{3'}$, $H^{5'}$, s	$H^{2''}$, s	$2H^{3''}$, $2H^{6''}$, s	$2H^{4''}$, $2H^{5''}$, s	$H^{1'''}$, $10H^{2'''-6'''}$, m
VIa	7.54	7.86–7.92	7.60 (^3J 9.2)	9.12 (^3J 7.4)	7.98	7.40				1.18
VIb	7.45	7.79–7.82	7.58 (^3J 9.0)	9.10 (^3J 7.4)	7.95	7.36				1.20
VIc	7.46	7.78–7.81	7.57 (^3J 9.2)	9.10	7.96	7.32				1.21
VId	7.46	7.78–7.81	7.57 (^3J 9.2)	9.10 (^3J 7.4)	7.94	7.36				1.22
VIe	7.47	7.80–7.84	7.59 (^3J 9.2)	9.14 (^3J 7.3)	7.92	7.92				1.20
VIf	7.46	7.81–7.85	7.58 (^3J 9.0)	9.10 (^3J 7.3)	7.94	7.30				1.18
VIIa	7.56	7.88–7.92	7.60 (^3J 9.2)	9.18 (^3J 7.2)	7.98	7.30	5.86	2.03	1.58	
VIIb	7.49	7.79–7.84	7.58 (^3J 9.0)	9.00 (^3J 7.2)	8.00	7.22	5.80	2.04	1.56	
VIIc	7.48	7.78–7.82	7.58 (^3J 9.0)	9.00 (^3J 7.3)	8.00	7.20	5.80	2.04	1.60	
VIId	7.48	7.79–7.83	7.58 (^3J 9.2)	9.00 (^3J 7.3)	7.94	7.37	5.80	2.00	1.60	
VIIe	7.46	7.80–7.83	7.64 (^3J 9.2)	9.14 (^3J 7.2)	7.96	7.96	5.72	2.06	1.58	
VIIf	7.48	7.80–7.84	7.60 (^3J 9.2)	9.02 (^3J 7.3)	7.93	7.28	5.75	2.02	1.54	
VIIg	7.48	7.78–7.82	7.57 (^3J 9.2)	9.12 (^3J 7.3)	7.98	7.24	5.84	2.06	1.56	
VIIh	7.46	7.76–7.81	7.60 (^3J 9.0)	9.12 (^3J 7.2)	7.94	7.88	5.82	2.04	1.59	

substituted benzo[*f*]quinolines **VIIa**–**VIIh** are displaced to the red region, and their intensity is larger, as compared to cyclohexane derivatives **VIa**–**VIh**; this is the result of conjugation between the cyclohexene C=C bond and the other part of the molecule.

The ^{1}H NMR spectra of benzo[*f*]quinolines **VI** and **VII** (Table 3) contain a signal at δ 7.45–7.56 ppm, which belongs to 2-H in the pyridine ring. Protons in the phenyl ring give a multiplet at δ 7.92–7.98 ppm and a signal at δ 7.20–7.40 ppm. Substituents in the phenyl ring (except for the nitro group) weakly affect the proton chemical shifts. The presence of a nitro group in the *para* position makes protons in the phenyl ring equivalent: they give a signal at δ 7.92 ppm in the spectrum of **VIe** and at δ 7.96 ppm in spectrum of **VIIe**. The signal at δ 1.18–1.21 ppm belongs to protons in the cyclohexane ring, and signals from methylene protons in the cyclohexene ring appear at δ 2.00–2.06 and 1.54–1.60 ppm. Unlike compounds **VI** and **VII**, dihydro derivatives **VIII** and **IX** display in the ^{1}H NMR spectra signals at δ 4.81 and 6.22 ppm, which belong to the NH and 3-H protons, respectively.

Benzo[*f*]quinoline derivatives are promising from the viewpoint of stabilization of polymeric materials against simultaneous action of temperature and radiation. Intermediate β-amino ketones of the naphthalene

series attract interest as potential biologically active substances and initial compounds for the synthesis of various oxygen- and nitrogen-containing organic compounds.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Tesla BS-567 spectrometer (100 MHz) from solutions in benzene-d_6 containing HMDS as internal reference. The IR spectra were obtained on a UR-20 instrument from samples prepared as KBr pellets. The UV spectra were measured on a Specord UV-Vis spectrophotometer. The melting points were determined on a Kofler device. Acetylcyclohexane (**I**) was synthesized by the procedure described in [13]. 1-Acetylcyclohexene (**II**) was obtained from cyclohexene and acetyl chloride according to [14].

***N*-Arylmethylene-2-naphthylamines IIIa–IIIh** were synthesized by heating equimolar amounts of the corresponding aldehyde and 2-naphthylamine in ethanol [17].

2-Aryl-2-(2-naphthylamino)ethyl cyclohexyl ketones IVa–IVf *(general procedure).* A solution of 10 mmol of Schiff base **IIIa–IIIf** and 10 mmol of acetylcyclohexane (**I**) in 40 ml of ethanol was heated to 60°C, and 0.06–0.07 ml of concentrated hydrochloric acid was added. After cooling, crystals precipitated from the solution. The product was neutralized with aqueous ammonia, filtered off, and purified by repeated crystallization from ethanol.

2-Aryl-2-(2-naphthylamino)ethyl 1-cyclohexenyl ketones Va–Vg were synthesized in a similar way using 1-acetylcyclohexene (**II**) and the corresponding Schiff base **IIIa–IIIg** on heating to 40°C.

3-Aryl-1-cyclohexylbenzo[*f*]quinolines VIa–VIh. A mixture of 10 mmol of acetylcyclohexane (**I**), 10 mmol of Schiff base **IIIa–IIIh**, 0.3–0.35 ml of concentrated hydrochloric acid, and 40 ml of toluene was heated for 4–5 h at 110°C. After cooling, the precipitate was filtered off, treated with aqueous ammonia, washed with ethanol, and recrystallized from ethanol–toluene (3 : 1).

3-Aryl-1-(1-cyclohexenyl)benzo[*f*]quinolines VIIa–VIIh were synthesized as described above for compounds **VIa–VIh** from methyl ketone **II** and Schiff bases **IIIa–IIIh** as initial compounds. After cooling, the precipitate (somewhat contaminated with tars) was filtered off, washed with ethanol, treated with aqueous ammonia, and purified by repeated crystallization from ethanol–toluene (4 : 1).

3-Aryl-1-cyclohexyl-3,4-dihydrobenzo[*f*]quinolines VIIIb, VIIIc, VIIIe, and VIIIf *(general procedure).* A solution of 15 mmol of acetylcyclohexane (**I**), 10 mmol of Schiff base **IIIb**, **IIIc**, **IIIe**, or **IIIf**, and 0.15 ml of concentrated hydrochloric acid in 50 ml of ethanol was heated for 4 h under reflux. The mixture was cooled, neutralized with aqueous ammonia, and evaporated by 1/4 of the initial volume. The precipitate was filtered off, washed with water and ethanol, and recrystallized from ethanol–nitromethane (1 : 1).

3-Aryl-1-(1-cyclohexenyl)-3,4-dihydrobenzo[*f*]quinolines IXc, IXe, and IXf *(general procedure).* 1-Acetylcyclohexene (**II**), 12 mmol, was added to a solution of 10 mmol of Schiff base **IIIc**, **IIIe**, or **IIIf** and 0.13 ml of concentrated hydrochloric acid in 50 ml of ethanol. The mixture was heated for 2.5 h under reflux and cooled, and the precipitate was filtered off, treated with aqueous ammonia, washed with ethanol, and recrystallized from ethanol–nitromethane (1 : 1).

Cyclization of amino ketones IVa–IVf and Va–Vg *(general procedure). a.* A solution of 5 mmol of amino ketone **IVa–IVf** or **Va–Vg** and 0.20–0.25 ml of concentrated hydrochloric acid in 25 ml of toluene was heated for 2.5–3 h at 110°C. The precipitate of benzo[*f*]quinoline **VIa–VIf** or **VIIa–VIIg** was filtered off, treated with aqueous ammonia, and recrystallized. Yield 56–72%.

b. A solution of 5 mmol of amino ketone **IVb, IVc, IVe, IVf, Vc, Ve**, or **Vf** and 0.20 ml of concentrated hydrochloric acid in 25 ml of ethanol was heated for 2–3 h under reflux. The products were isolated as described above for the synthesis of dihydrobenzo[*f*]-quinolines **VIII** and **IX**. Yield 29–40%.

Dehydrogenation of 3,4-dihydrobenzo[*f*]quinolines VIIIb, VIIIc, VIIIe, VIIIf, IXc, IXe, and IXf *(general procedure).* A solution of 5 mmol of dihydrobenzoquinoline **VIIIb**, **VIIIc**, **VIIIe**, **VIIIf**, **IXc**, **IXe**, or **IXf** and 0.15–0.20 ml of concentrated hydrochloric acid in 25 ml of ethanol was heated for 2–2.5 h under reflux. The products were isolated as described above for the synthesis of benzo[*f*]quinolines **VI** and **VII**. Yield 56–74%.

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