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Synthesis of New Haloderivatives of Benzo[*f*]quinoline and 4,7-Phenanthroline

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Abstract—By condensations of 2-naphthylamine and 6-aminoquinoline with halogen-substituted benzaldehydes, 3-acetylpyridine and acetophenone derivatives new 1,3-diaryl(heteryl)benzo-[*f*]quinolines and 4,7-phenanthrolines were synthesized containing atoms of fluorine, bromine, and chlorine in the phenyl rings.

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The introduction of a halogen atom into a molecule of a heterocyclic compound leads to extension of its synthetic opportunities and of the range of its physiological action [1, 2].

The published information on the synthesis of chlorine-substituted benzo[*f*]quinolines and 4,7-phenanthrolines [2–4] concerned the substitution of hydroxy group by the treatment of $POCl_3$ and PCl_5 of the corresponding hydroxy derivatives prepared along Conrad–Limpach and Knorr reactions from 2-naphthylamine, 6-aminoquinoline, or *p*-phenylenediamine and esters of β-oxo and β-dicarboxylic acids. The direct halogenation of azaphenanthrenes requires stringent conditions due to hindering the electrophilic substitution. The bromination of 4,7-phenanthroline is carried out in oleum and results in an inseparable mixture of mono-, di-, and tribromo derivatives [5].

The three-component condensation of arylamines, aldehydes, and CH-acids that we have developed for the synthesis of polyfused azaaromatic compounds [6–8] provides a possibility to introduce halogen atoms into the molecule of the heterocycle simultaneously with the formation of the azaphenanthrene framework when synthons contain halogen atoms. The variation of substituents in the molecules of initial reagents makes it possible to introduce into the azaphenanthrene structure both identical and different halogen atoms, and also to obtain compounds containing alongside the halogen another pharmacophore atoms and groups.

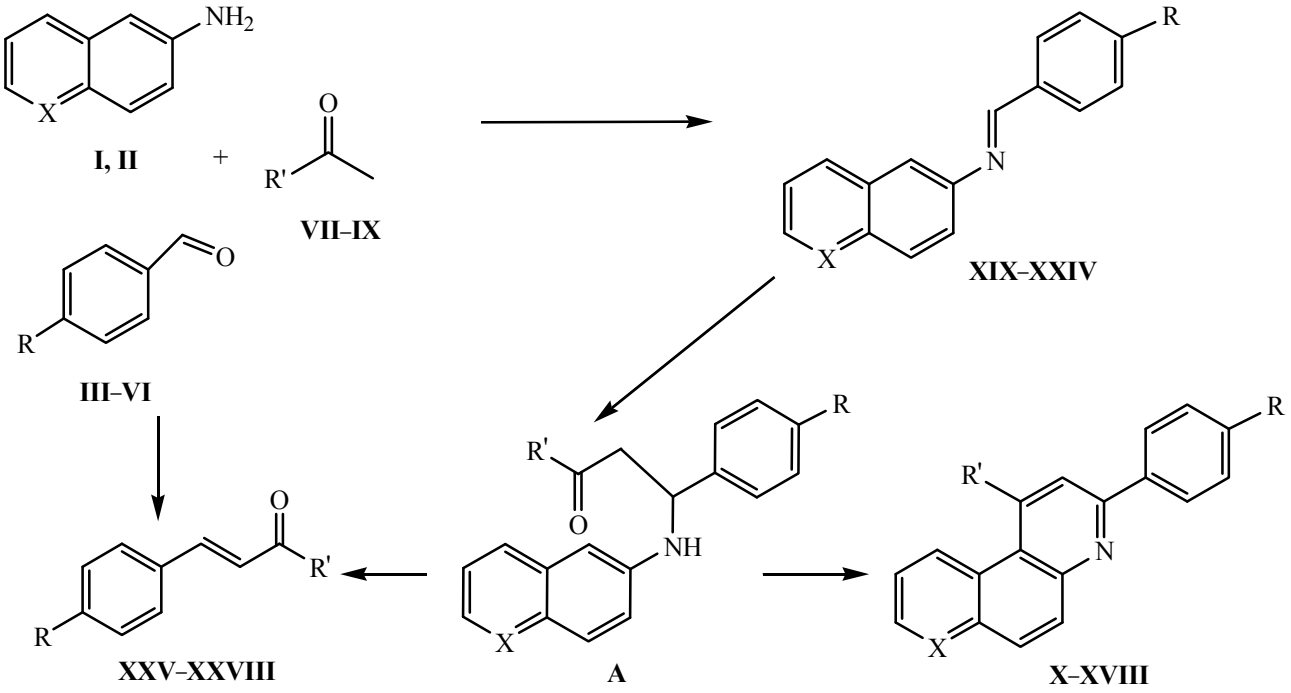
In this research we for the first time fulfilled the synthesis of halosubstituted benzo[*f*]quinolines and 4,7-phenanthrolines proceeding from 2-naphthylamine (**I**), 6-aminoquinoline (**II**), 4-(2-fluorobenzyloxy)-, *p*-fluoro-, *p*-chloro-, *p*-bromobenzaldehyde (**III–VI**), *p*-chloro-, *p*-bromoacetophenone, and 3-acetylpyridine (**VII–IX**).

The condensation of amines **I, II**, aldehydes **III–VI**, and methyl ketones **VII–IX** was carried out by boiling for 4–6 h of a solution of equimolar amounts of the reagents in 1-butanol in the presence of concn. HCl as a catalyst. In 38–55% yield we obtained previously unknown 1,3-diarylbenzo[*f*]quinolines **X, XI** and 1,3-diaryl-4,7-phenanthrolines **XII–XVI** containing halogen atoms in phenyl substituents, and also fluoro derivatives of 3-aryl-1-(3-pyridyl)-benzo[*f*]quinoline **XVII, XVIII**.

As follows from the results of our previous investigations [6–8] the reaction proceeds through the stage of azomethine **XIX–XXIV** formation that further reacts with the methyl ketone along the known mehanism [9] involving the addition of the CH-acid to the C=N bond of the azomethine and dehydrocyclization of the obtained adduct **A** into benzo[*f*]quinoline **X, XI, XVII, XVIII** or 4,7-phenanthroline **XII–XVI** respectively. In the presence of HCl formed aza- and diazaphenanthrene hydrochlorides that on treating with ammonium hydroxide furnished free bases **X–XVIII**.

To prove the involvement of azomethines in the formation of the azaphenanthrene structure we carried out the condensation of amines **I, II** with aldehydes **III–**

Scheme.



R = F (**III, XII, XVII, XIX, XXII, XXV**), Cl (**IV, X, XI, XIII, XV, XX, XXIII, XXVI**), Br (**V, XIV, XVI, XXIV, XXVII, XXVIII**), $OCH_2C_6H_4$-2-F (**VI, XVIII, XXI**); R' = 4-ClC_6H_4 (**VII, X, XI, XIII, XV, XXVI, XXVII**), 4-BrC_6H_4 (**VIII, XI, XV, XVI, XXVIII**), 3-pyridyl (**IX, XVII, XVIII, XXV**); X = CH (**I, X, XI, XVII, XVIII–XXI**), N (**II, XII–XVI, XXII–XXIV**).

VI, and the obtained Schiff bases **XIX–XXIV** were brought into the reaction with acetophenones **VII, VIII** or with 3-acetylpyridine (**IX**) under the above described conditions. The corresponding benzoquinolines **X, XI, XVII, XVIII** and phenanthrolines **XII–XVI** were obtained in 40–56% yield.

We failed to isolate intermediate aminoketones **A**. Under mild conditions commonly applied in the synthesis of aminoketones [8–10] (room temperature or hetating the reagents in ethanol without catalyst), the reaction stopped at the stage of azomethines formation. At boiling the reagents in ethanol in the presence of concn. HCl we isolated halosubstituted chalcones **XXV–XXVIII** formed by the competing aldol condensation of the acetophenone with the aromatic aldehyde directly brought into the reaction at the three-component condensation or arising as a retrodecomposition product of the Schiff base. Chalcones **XXV–XXVIII** may also form by deamination of intermediate aminoketone **A**. In 1-butanolε evidently both the hydrolysis of azomethines and cleavage of aminoketones are hampered, therefore the condensation occurs with the prevailing formation of benzo[*f*]quinolines and 4,7-phenanthrolines.

It should be noted that even in 1-butanol we did not always prevented the chalcones formation. Minor quantities of compounds **XXVI–XXVIII** were isolated alongside the target 4,7-phenanthrolines **XIII, XIV**, **XVI** in the reactions of acetophenones **VII, VIII** both with arylmethylene-6-aminoquinolines **XXIII, XXIV** and 6-aminoquinoline (**II**) and aldehydes **IV, V** evidently because the nucleophilic activity of the amino group was decreased and the reactivity of 6-aminoquinoline was diminished compared to the 2-naphthylamine due to the effect of the electronegative nitrogen atom of the quinoline ring in amine **II**, and also because of decreased polarization and the chemical activity of the azomethine bond. Only in the case of *p*-fluoro substituent that to the highest extent activated the carbonyl group of aldehyde **III** and the azomethine bond in the molecule of Schiff base **XXII** owing to the *–I*-effect 4,7-phenanthroline **XII** formed selectively with the maximum yield (54%) for phenanthrolines.

The structure of methyl ketone **VII–IX** weakly affects the course of the condensation. Haloacetophenones **VII, VIII** and 3-acetylpyridine (**IX**) in reaction with 2-naphthylamine (**I**) and arylaldehydes **III, IV, VI** afforded

practically the same yield of benzo[*f*]quinolines.

In the IR spectra of compounds **X–XVIII** the stretching vibrations of the CH bonds of the aromatic rings were observed at 3060–3030 cm^{-1}. The bands at 720–715 and 840–830 cm^{-1} insensitive to the substituent effects belong to the out-of-plane bending vibrations of the two contiguous C–H bonds at atoms C^5 and C^6 of the azaphenanthrene skeleton [11]. Compounds **XII, XVII, XVIII** possess a strong absorption band at 1155 cm^{-1} belonging to the stretching vibrations of the C–F bond, azaphenanthrenes **X–XV**, a band at 855–850 cm^{-1}, corresponding to the stretching vibrations of C–Cl bonds, compounds **XI, XIV–XVI**, a band of the stretching vibrations of the C–Br bond at 575–570 cm^{-1}. The IR spectrum of benzoquinoline **XVIII** also contained strong bands at 2870 and 1235 cm^{-1} originating from the stretching vibrations of the bonds of CH_2 and C–O–C groups respectively.

In the mass spectra of aza- and diazaphenanthrenes **X–XVIII** the molecular ion peak $[M]^+$ is the most abundant, the other observed peaks are $[M - H]^+$ (I_{rel} 65–84%) and weak peaks of ions $[M - HlgC_6H_4]^+$ and $[M - HCN]^+$ (I_{rel} 5–14%). Also the peaks of double-charged molecular ions $[M]^{2+}$ and $[M - HCN]^{2+}$ are present characteristic of fused heteroaromatic compounds.

The general pattern of the UV absorption spectra of benzoquinolines and 4,7-phenanthrolines **X–XVIII** is characteristic of compounds of angular structure [12, 13]. The bands present in the spectra (220–261, 288–296, 333–360 nm) corresponding to the π–π*-electron transitions in the fused aromatic compounds are interpreted as Clar's β-, *p*-, and α-bands [14]. As compared to phenanthrene [12] in the spectra of compounds **X–XVIII** the vibronic structure is smoothed, and the intensity of the α-band decreased, the most obvious in the spectra of phenanthrolines **XII–XVI** apparently due to the accumulation of nitrogen atoms in the system. The halogen atoms in the phenyl substituents and also the replacement of the aryl substituent in the position *1* of the azaphenanthrene framework by a pyridine materially does not affect the position and the intensity of the absorption bands.

In the ^{1}H NMR spectra of benzoquinolines **X, XI, XVII, XVIII** signals of the aromatic protons are observed in the range 7.08– 8.80 ppm. Among them it is possible to distinguish the pairs of two-proton doublets corresponding to halosubstituted benzene rings and a singlet of H^2 proton of 1,3-disubstituted benzoquinoline ring. The spectrum of compound **XVIII** contains a singlet of methylene protons of the 2-fluorobenzoxyphenyl substituent at 5.36 ppm. In the spectra of 1,3-diaryl-4,7-phenanthrolines **XII–XVI** owing to the presence in the phenanthrene skeleton of two symmetrically placed nitrogen atoms it was possible to assign the proton signals of the phenanthroline fragment. The spectra of compounds **XII–XVI** contained two characteristic doublets at in the 8.23–8.36 ppm (^3J 9.0–9.2 Hz) belonging to the signals of protons H^5 and H^6 of phenanthroline. These signals and also the signals of protons $H^{2,8\text{-}10}$ are not subjected to the anisotropic effect of halogen atoms, only in the spectrum of the fluoro-substituted phenanthroline **XII** a downfield shift is observed for a contiguous to the phenyl H^2 proton due to the strong –*I*-effect of the fluorine atom. At the same time in the spectra of 1,3-diaryl-4,7-phenanthrolines **XII–XVI** compared to the previously investigated spectra of the fused 4,7-phenanthroline derivatives [10] a strong upfield shift was observed for the signal of H^{10} proton occurring in the area of the action of the ring current of the aromatic ring located in the position *1* of the phenanthroline system.

EXPERIMENTAL

IR spectra were recorded on a Fourier spectrophotometer Nicolet Protйgй-460 from pellets with KBr. Mass spectra were measured on an instrument Finnigan MAT INCOS 50 at ionizing electrons energy 70 eV and on GC-MS spectrometer Hewlett-Packard 5890/5972 in an electron impact mode at the energy 70 eV, column HP-5MS [30 m × 0.25 mm, film of the stationary phase 0.25 μm thick (5% PLMe Silicone)], vaporizer temperature 250°C. UV spectra were taken from solutions of compounds in ethanol (C 10^{-4} mol l^{-1}) on a spectrophotometer Specord UV-Vis. NMR spectra were registered on spectrometers Bruker AC-500 (500 MHz) and Tesla BS-567 (100 MHz) from solutions in deuterochloroform, internal reference TMS.

Melting points were measured on a Koeffler heating block.

Aryl(or 3-pyridyl)methylene-2-naphthyl- (or 6-quinolyl)amines XIX–XXIV were prepared by procedure [15]. 1-(3-Pyridyl)-3-(4-fluorophenyl)-propenone (**XXV**) and 1,3-diarylpropenones **XXVI–XXVIII** were identified by melting points, IR and mass spectra [16–18].

1,3-Diaryl- and 1-(3-pyridyl)-3-arylbenzo[*f*]-quinolines (X, XI, XVII, XVIII). *a*. A mixture of

5 mmol of 2-naphthylamine (**I**), 5 mmol of aldehyde **III, IV, VI**, 5 mmol of methyl ketone **VII–IX**, 20 ml of 1-butanol, and 0.5 ml of concn. HCl was boiled for 2.5 h. The precipitate separated on cooling was filtered off, neutralized with 25% aqueous NH_4OH, and washed with water. Compounds **X, XI, XVII** were recrystallized from a mixture ethanol–benzene, 2:1, benzoquinoline **XVIII**, from methanol.

b. A solution of 5 mmol of azomethine **XIX–XXI**, 5 mmol of ketone **VII–IX**, 20 ml of 1-butanol, and 0.5 ml of concn. HCl was boiled for 2 h. Reaction products **X, XI, XVII, XVIII** were isolated as described above.

1,3-Di(4-chlorophenyl)benzo[*f*]quinoline (X). Yield 50 (*a*), 53% (*b*), mp 201–202°C. UV spectrum, λ_{max}, nm (log ε): 220 (4.60), 257 (4.62), 290 (4.81), 333 (4.00), 356 (3.87). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.08–7.52 m (4H, H_{arom}), 7.58 d (2H, H_{arom}, ^3J 8.0), 7.67 d (2H, H_{arom}, ^3J 8.0), 7.74 s (1H, H^2), 7.98 d (2H, H_{arom}, ^3J 8.0), 8.14 m (4H, H_{arom}). Found, %: C 74.85; H 3.69; Cl 17.53; N 3.35. $C_{25}H_{15}Cl_2N$. Calculated, %: C 75.00; H 3.75; Cl 17.75; N 3.50.

1-(4-Bromophenyl)-3-(4-chlorophenyl)benzo[*f*]quinoline (XI). Yield 55 (*a*), 56% (*b*), mp 206–207°C. UV spectrum, λ_{max}, nm (log ε): 222 (4.56), 255 (4.60), 288 (4.80), 335 (3.97), 354 (3.89). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.13–7.50 m (4H, H_{arom}), 7.61 d (2H, H_{arom}, ^3J 8.3), 7.68 d (2H, H_{arom}, ^3J 8.3), 7.73 s (1H, H^2), 8.00 d (2H, H_{arom}, ^3J 8.3), 8.19 m (4H, H_{arom}). Found, %: C 67.32; H 3.25; Br 17.85; Cl 7.81; N 3.03. $C_{25}H_{15}BrClN$. Calculated, %: C 67.49; H 3.37; Br 18.00; Cl 7.99; N 3.15.

1-(3-Pyridyl)-3-(4-fluorophenyl)benzo[*f*]quinoline (XVII). Yield 51 (*a*), 55% (*b*), mp 124–125°C. UV spectrum, λ_{max}, nm (log ε): 220 (4.51), 253 (4.59), 289 (4.82), 336 (3.95), 356 (3.84). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.23–7.49 m (4H, H_{arom}), 7.53–7.68 m (4H, H_{arom}), 7.75 s (1H, H^2), 7.95–8.10 m (4H, H_{arom}), 8.62–8.80 m (2H, H_{arom}). Found, %: N 7.82. $C_{24}H_{15}FN_2$. Calculated, %: N 8.00.

1-(3-Pyridyl)-3-[4-(2-fluorobenzyloxy)phenyl]benzo[*f*]quinoline (XVIII). Yield 52 (*a*), 53% (*b*), mp 183–184°C. UV spectrum, λ_{max}, nm (log ε): 225 (4.52), 258 (4.60), 291 (4.83), 335 (3.99), 358 (3.89). ^{1}H NMR spectrum, δ, ppm: 5.36 C (2H, OCH_2), 7.06 m (5H, H_{arom}), 7.45 m (4H, H_{arom}), 7.70 s (1H, H^2), 7.90 m (2H, H_{arom}), 8.10 m (3H, H_{arom}), 8.25 d (2H, H_{arom}, ^3J 8.1 Hz), 8.80 m (2H, H_{arom}). Found, %: N 5.95. $C_{31}H_{21}FN_2O$. Calculated, %: N 6.14.

1,3-Diaryl-4,7-phenanthrolines XII–XVI. A solution of equimolar amounts (5 mmol) of 6-aminoquinoline (**II**), of an appropriate aldehyde **III–V** and haloacetophenone **VII, VIII** (method *a*) or of azomethine **XXII–XXIV** and ketone **VII, VIII** (method *b*) in 20 ml of 1-butanol with 0.5 ml of concn. HCl was boiled for 2–2.5 h. The precipitate separated on cooling was filtered off, neutralized with 25% aqueous NH_4OH, and washed with water. Phenanthroline **XII** was recrystallized from ethanol, compounds **XIII–XVI**, from a mixture ethanol–benzene, 2:1.

3-(4-Fluorophenyl)-1-(4-chlorophenyl)-4,7-phenanthroline (XII). Yield 51 (*a*), 54% (*b*), mp 190–191°C. UV spectrum, λ_{max}, nm (log ε): 225 (4.62), 253 (4.60), 291 (4.80), 336 (3.90), 356 (3.71). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.20 d.d (1H, H^9, ^3J 8.7, ^4J 4.0), 7.40 d, 7.52 d, 7.65 d, 8.20 d (8H, H_{arom}, ^3J 8.1), 7.92 s (1H, H^2), 7.96 d (1H, H^{10}, ^3J 8.7). 8.27 d, 8.36 d (2H, $H^{5,6}$, ^3J 9.2), 8.86 d.d (1H, H^8, ^3J 4.0, ^4J 1.8), Found, %: N 7.09. $C_{24}H_{14}ClFN_2$. Calculated, %: N 7.28.

1,3-Di(4-chlorophenyl)-4,7-phenanthroline (XIII). Yield 41 (*a*), 43% (*b*), mp 234–235°C. UV spectrum, λ_{max}, nm (log ε): 226 (4.63), 259 (4.64), 296 (4.78), 338 (3.88), 355 (3.60). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.15 d.d (1H, H^9, ^3J 8.8, ^4J 4.2), 7.44 d, 7.51 d, 7.58 d, 8.18 d (8H, H_{arom}, ^3J 8.8), 7.76 s (1H, H^2), 7.95 d (1H, H^{10}, ^3J 8.8), 8.26 d, 8.34 d (2H, $H^{5,6}$, ^3J 9.0), 8.87 d.d (1H, H^8, ^3J 4.2, ^4J 1.7). Found, %: C 71.64; H 3.41; Cl 17.59; N 6.75. $C_{24}H_{14}Cl_2N_2$. Calculated, %: C 71.82; H 3.49; Cl 17.71; N 6.98.

3-(4-Bromophenyl)-1-(4-chlorophenyl)-4,7-phenanthroline (XIV). Yield 38 (*a*), 40% (*b*), mp 237–238°C. UV spectrum, λ_{max}, nm (log ε): 227 (4.68), 261 (4.67), 296 (4.84), 339 (3.86), 360 (3.65). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.17 d.d (1H, H^9, ^3J 8.3, ^4J 4.3), 7.44 d, 7.59 d, 7.68 d, 8.14 d (8H, H_{arom}, ^3J 8.8), 7.78 s (1H, H^2), 7.97 d (1H, H^{10}, ^3J 8.3), 8.27 d, 8.35 d (2H, $H^{5,6}$, ^3J 9.1), 8.88 d.d (1H, H^8, ^3J 4.3, ^4J 1.8). Found, %: C 64.56; H 3.01; Br 17.63; Cl 7.75; N 6.18. $C_{24}H_{14}BrClN_2$. Calculated, %: C 64.65; H 3.14; Br 17.96; Cl 7.97; N 6.29.

1-(4-Bromophenyl)-3-(4-chlorophenyl)-4,7-phenanthroline (XV). Yield 43 (*a*), 44% (*b*), mp 229–230°C. UV spectrum, λ_{max}, nm (log ε): 221 (4.60), 258 (4.59), 293 (4.72), 337 (3.90), 352 (3.75). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.14 d.d (1H, H^9, ^3J 8.1, ^4J 4.0), 7.42 d, 7.56 d, 7.60 d, 8.10 d (8H, H_{arom}, ^3J 8.6),

7.72 s (1H, H^2), 7.95 d (1H, H^{10}, ^3J 8.1), 8.23 d, 8.31 d (2H, $H^{5,6}$, ^3J 9.0), 8.81 d.d (1H, H^8, ^3J 4.0, ^4J 1.9). Found, %: C 64.48; H 3.09; Br 17.71; Cl 7.68; N 6.14. $C_{24}H_{14}BrClN_2$. Calculated, %: C 64.65; H 3.14; Br 17.96; Cl 7.97; N 6.29.

1,3-Di(4-bromophenyl)-4,7-phenanthroline (XVI). Yield 40 (*a*), 42% (*b*), mp 240–241°C. UV spectrum, λ_{max}, nm (log ε): 223 (4.58), 257 (4.57), 295 (4.79), 337 (3.84), 356 (3.69). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.16 d.d (1H, H^9, ^3J 8.6, ^4J 4.3), 7.38 d, 7.52 d, 7.61 d, 8.19 d (8H, H_{arom}, ^3J 8.4), 7.74 s (1H, H^2), 7.98 d (1H, H^{10}, ^3J 8.6), 8.25 d, 8.34 d (2H, $H^{5,6}$, ^3J 9.1), 8.83 d.d (1H, H^8, ^3J 4.3, ^4J 2.1). Found, %: C 58.53; H 2.80; Br 32.44; N 5.59. $C_{24}H_{14}Br_2N_2$. Calculated, %: C 58.78; H 2.86; Br 32.65; N 5.71.

1-(3-Pyridyl)-3-(4-fluorophenyl)propenone (XXV) was obtained by the procedure described for benzoquinolines **X**, **XI**, **XVII**, **XVIII**, not in 1-butanol but in ethanol. The solution was evaporated to 1/2 of its volume, the separated precipitate was filtered off and recrystallized from ethanol. Yield 62%, mp 120–121°C (mp 122°C [16]). IR spectrum, ν, cm^{-1}: 1660 (CO), 1590 (CH=CH). Mass spectrum, *m/z* (I_{rel}, %): 227 $[M]^+$ (100). $C_{14}H_{10}FNO$. Calculated *M* 227.

1,3-Diarylpropenones XXVI–XXVIII were obtained by the procedure described for compounds **XIII–XVI** and were separated by evaporation of the mother liquor after the isolation of the precipitates of phenanthrolines **XIII, XIV, XVI** hydrochlorides. Compounds **XXVI**, **XXVII** were recrystallized from ethanol, propenone **XXVIII**, from a mixture ethanol–benzene, 3:1.

1,3-Di(4-chlorophenyl)propenone (XXVI). Yield 8%, mp 155–156°C (mp 155–157°C [17]). IR spectrum, ν, cm^{-1}: 1660 (CO), 1595 (CH=CH). Mass spectrum, *m/z* (I_{rel}, %): 277 $[M]^+$ (100). $C_{15}H_{10}Cl_2O$. Calculated *M* 277.

1-(4-Bromophenyl)-3-(4-chlorophenyl)propenone (XXVII). Yield 10%, mp 172–173°C (mp 173°C [18]). IR spectrum, ν, cm^{-1}: 1665 (CO), 1600 (CH=CH). Mass spectrum, *m/z* (I_{rel}, %): 321 $[M]^+$ (100). $C_{15}H_{10}BrClO$. Calculated *M* 321.

1,3-Di(4-bromophenyl)propenone (XXVIII). Yield 11%, mp 195–196°C (mp 194–196°C [17]). IR spectrum, ν, cm^{-1}: 1670 (CO), 1605 (CH=CH). Mass spectrum, *m/z* (I_{rel}, %): 366 $[M]^+$ (100). $C_{15}H_{10}Br_2O$. Calculated *M* 366.

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