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Condensation of N-Monosubstituted 2-Naphthylamines, Formaldehyde, and Cyclic β-Diketones. One-Pot Synthesis of 2,4-Disubstituted Derivatives of 1,2,3,4-Tetrahydrobenzo[*f*]quinoline

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Abstract—Various spirocyclic derivatives of 1,2,3,4-tetrahydrobenzo[*f*]quinoline containing substituents in the positions *2* and *4* of the ring were obtained by one-pot multicomponent condensation of available *N*-benzyl-2-naphthylamines, formaldehyde, and cyclic β-diketones of cyclohexanedione series.

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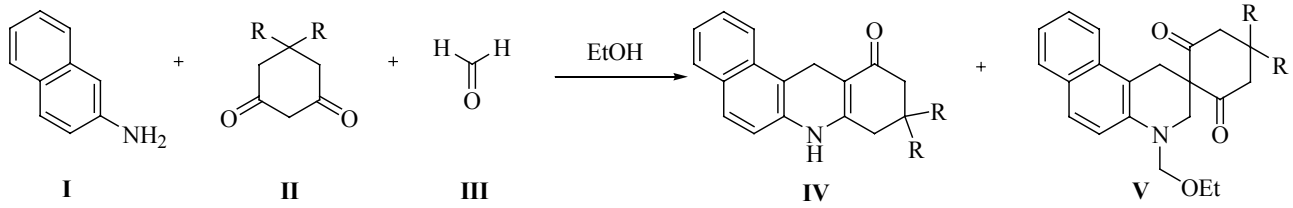
Compounds of the benzoquinoline series exhibit a wide range of biologic action, and this stimulates the synthesis and investigation of new compounds of this class [1]. Various substituted benzo[*f*]quinolines possess antibacterial [2] and enzyme-inhibiting [3, 4] properties, block the dopamine receptors [5], and find application in the other fields of biological and pharmaceutical chemistry [6]. Besides the benzo[*f*]quinoline framework underlies the structure of the ergoline alkaloids group of ergot *Claviceps purpurea* [7]. The majority of the biologically active benzo[*f*]quinolines contain substituents in the positions *2* and *4* of the ring, but only a small number of methods of selective syntheses exists for the preparation of benzo[*f*]quinoline derivatives with these substituents, and the 2,4-disubstituted benzo[*f*]quinolines remain relatively difficultly accessible compounds.

Formerly in the research on the three-component condensation of aromatic amines with cyclic β-diketones and various aldehydes we demonstrated that the reaction of 2-naphthylamine (**I**) with dimedone (**II**, R = CH_3) in the presence of formaldehyde (**III**) alongside with the "classic" product of the benzo[*a*]acridone structure **IV** provided a small quantity of spirocyclic benzo[*f*]quinoline **V** containing substituents in the positions *2*, *4* of the ring (Scheme 1) [8].

We assumed that the formation of the mixture of benzo[*a*]acridone **IV** and benzo[*f*]quinoline **V** proceeded probably through the following transformations. The reaction of the initial β-diketone with the formaldehyde along the mechanism of Knoevenagel reaction results in the formation of an α,β-unsaturated diketone **A**, where the double bond is activated by the conjugation with two

Scheme 1.



I **II** **III** **IV** **V**

carbonyl groups. 2-Naphthylamine (**I**) added the molecule of diketone **A** providing aminodiketone **B**, which through an intramolecular attack of the primary amino group on the carbon atom of the enolized ketone carbonyl of the β-diketone fragment of the molecule underwent the cyclization into intermediate **C.** The elimination of water molecule afforded benzo[*a*]-acridone **IV**.

The formation of the heterocyclic framework of benzoquinoline compound **V** starts by the N-alkoxymethylation of the initial 2-naphthylamine to form N,O-acetal **D**. The aromatic ring of compound **D** added further the molecule of α,β-unsaturated ketone **A** forming aminodiketone **E** containing a secondary amino group. Aminodiketone **E** evidently did not underwent the direct cyclization due to less steric accessibility of the secondary amino group but by the intermolecular Mannich reaction througn an iminium intermediate **F** converted into a spirocyclic benzo[*f*]quinoline derivative **V** (Scheme 2). The low steric requirements of the small formaldehyde molecule and the high nucleophilicity of the secondary amino group govern the thermodynamic and steric probability of this reaction.

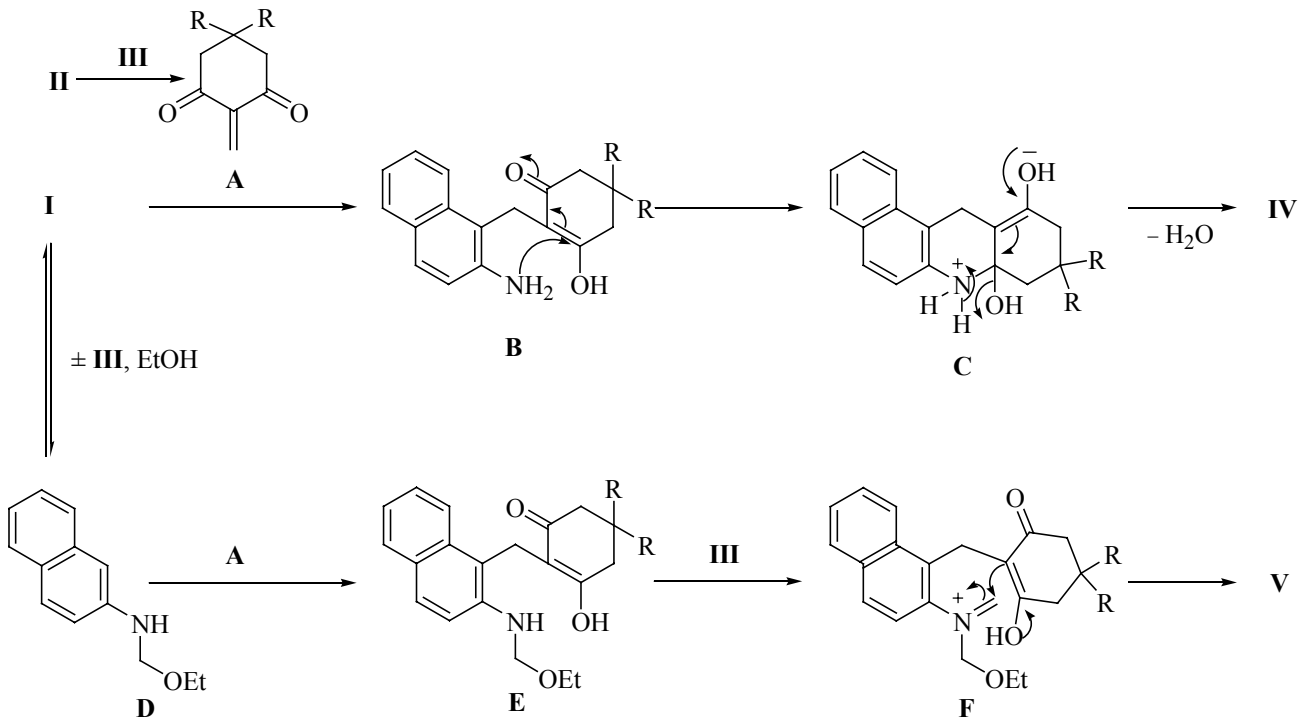
It follows from the suggested mechanism that at the use in the three-component condensation with the cyclic β-diketones and the formaldehyde of secondary amines as the amine component, in particular, of N-monosubstituted 2-naphthylamines formally analogous to compound **D**, the reaction should result prevailingly or selectively in the formation of spirocyclic benzo[*f*]quinoline derivatives similar to compound **V**. We performed a synthesis of a series of available *N*-benzyl-2-naphthylamines by reduction with sodium borohydride of Schiff bases obtained from 2-naphthylamine and appropriate aromatic aldehydes and brought them into the condensation with the formaldehyde and β-diketones of the cyclohexanedione series.

The condensation was performed by short boiling of a mixture of N-benzylamine **VIa–VIe**, excess formaldehyde (**III**), and 1,3-cyclohexanedione (**IIa**) or dimedone (**IIb**) in ethanol. The reaction products actually were exclusively spirocyclic derivatives of *N*-benzylbenzo[*f*]-quinoline **VIIa–VIIj** (Scheme 3). The reaction does not require a catalyst and virtually quantitatively yields individual compounds that precipitate from the reaction mixture, which maximally simplifies their isolation and purification.

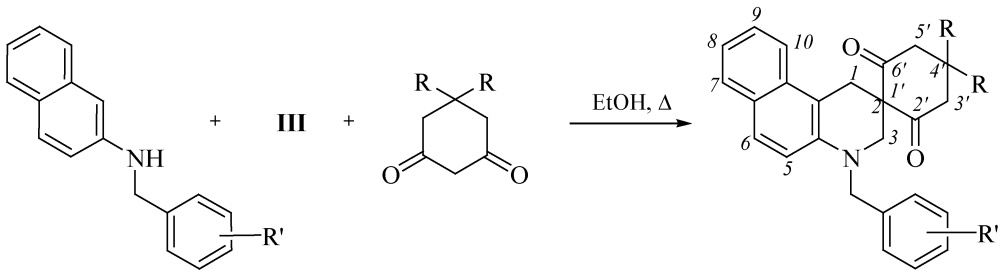
The structure of compounds obtained was established from their IR and ^{1}H NMR spectra.

IR spectra of compounds **VIIa–VIIj** contain two strong absorption bands of the carbonyl group at ~ 1680 and 1730 cm^{-1}. The presence of two absorption bands in the carbonyl region is characteristic of the nonenolizible

Scheme 2.



Scheme 3.



VIa–VIe **IIa, IIb** **VIIa–VIIk**

II, R = H (**a**), CH_3 (**b**); **VI**, R' = 4-OCH_3 (**a**), 3,4-$(OCH_3)_2$ (**b**), 3,4-OCH_2O (**c**), 4-Ph (**d**), 4-$CH(CH_3)_2$ (**e**); **VII**, R = H, R' = 4-OCH_3 (**a**), 3,4-$(OCH_3)_2$ (**b**), 3,4-OCH_2O (**c**), 4-Ph (**d**), $CH(CH_3)_2$ (**e**); R = CH_3, R' = 4-OCH_3 (**f**), 3,4-$(OCH_3)_2$ (**g**), 3,4-OCH_2O (**h**), 4-Ph (**i**), $CH(CH_3)_2$ (**j**).

dimedone ketoform, in particular, of 2,2-disubstituted dimedone derivatives [9]. The stretching vibrations of alkyl groups and cycloaliphatic C–H bonds give rise to the absorption in the region 3000–2800 cm^{-1}, C–H bonds in the aromatic ring, at 3100–3000 cm^{-1}. The fragments C–O–C present in the molecules of compounds **VIIa–VIIc**, **VIIf–VIIh** appear as bands in the region 1260–1220 cm^{-1}.

In the ^1H NMR spectra of compounds **VIIa–VIIj** characteristic signals were observed corresponding to substituents R' and a set of signals with the appropriate overall intensity in the region 6.7– 8.0 ppm belonging to the aromatic protons.

The geminal protons at the atom C^1 are enantiotopic and appear as a singlet with 2H integral intensity in the region 3.4–3.6 ppm. The protons at atom C^3 also appear as a singlet with 2H integral intensity in the region 3.5–3.7 ppm, the downfield shift of the signal is due to the deshielding effect of the contiguous nitrogen atom. The enantiotopic protons of the NCH_2Ar fragment suffer the deshielding influence of the nighboring nitrogen atom and the aromatic ring and thus appear in the spectra even more downfield (4.5–4.7 ppm) as a singlet with 2H integral intensity.

In the ^1H NMR spectra of compounds **VIIa–VIIe** synthesized with the use of 1,3-cyclohexanedione the protons at the atom $C^{4'}$ give rise to a broad multiplet of 2H integral intensity in the region 1.6–2.3 ppm. The protons at atoms $C^{3'}$ and $C^{5'}$ appear as a multiplet with the integral intensity of 4H at 2.5–2.9 ppm.

In the ^1H NMR spectra of compounds **VIIf–VIIj** obtained with the use of dimedone two diastereotopic methyl groups at the atom C^4' are observed as two three-proton singlets in the region 0.9–1.1 ppm. The pairs of protons at atoms C^3' and C^5'are mutually enantiotopic and give one signal in the spectrum, but inside each pair the protons are diastereotopic, and the signal is split due to the geminal coupling (*AC* system). As a result the spectra contain a characteristic for the *AC* system a pair of doublets with the "roof effect" and the coupling constant of 16 Hz and overall integral intensity 4H in the region 2.3–2.8 ppm.

Beside the 2,4-substituted benzoquinoline ring the compounds **VIIa–VIIj** contain a 2-azaspiro[5.5]undecene fragment that underlies the structure of a group of piperidine alkaloids (sibirin, nitramin, nitrabirin, etc.) isolated from *Nitraria sibirica Pall* and structurally like the neurotoxic alkaloids of the family of histrionicotoxins [10–12]. The partially decomposed 2-azaspiro[5.5]undecene system is also a structural fragment of some toxins isolated from some species of sea oysters [13].

Thus we developed a new simple selective method of synthesis of 2,4-disubstituted derivatives of 1,2,3,4-tetrahydrobenzo[*f*]quinoline based on the multicomponent condensation of available N-benzyl-2-naphthylamines with formaldehyde and cyclic β-diketones of cyclohexanedione series.

EXPERIMENTAL

IR spectra were recorded on a Fourier spectrophotometer Nikolet Protégé-460 from pellets with KBr. ^1H NMR spectra were registered on a spectrometer Tesla BS-567 (100 MHz) from solutions in $CDCl_3$, internal reference TMS. Melting points were measured on a Koeffler heating block.

***N*-Benzyl-2-naphthylamines VIa–VIe.** A mixture of

1.43 g (0.01 mol) of 2-naphthylamine and 0.012 mol of aromatic aldehyde in 100 ml of ethanol was boiled for 2 h. To a cooled solution was added 1 g (0.025 mol) of sodium borohydride. The mixture obtained was boiled for 3 h, another 1 g (0.025 mol) of sodium borohydride was added, and the boiling was continued for 3 h more. On cooling the reaction mixture was quenched by adding excess 20% water solution of NaOH. The amine precipitate was filtered off, washed with water, dried, and recrystallized from ethanol. Yield 70–85%. Analytical characteristics of amines obtained by this procedure were consistent with those of previously synthesized [14].

4-Benzyl-1,4-dihydro-3*H*-spiro[benzo[*f*]-quinoline-2,1'-cyclohexane]-2',6'-diones VIIa–VIIe. A mixture of 0.001 mol of aminea **VIa–VIe** and 0.12 g (0.004 mol) of paraformaldehyde in 25 ml of ethanol was boiled till complete dissolution of both components (10–15 min). To the obtained solution was added 0.112 g (0.001 mol) of 1,3-cyclohexanedione (**IIa**), the mixture was boiled for 10 min, cooled, the separated precipitate was filtered off and washed with ethanol (2×5 ml). The mother liquor was left standing in air till the major part of the solvent evaporated, and then the second portion of the product was filtered off.

4-(4-Methoxybenzyl)-1,4-dihydro-3*H*-spiro[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-dione (VIIa). Yield 91%, mp 182°C. IR spectrum, ν, cm $^{-1}$: 3000–3100 (C–H_{arom}), 2800–2960 (C–$H_{aliphat}$), 1726 (C=O), 1699 (C=O), 1235 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 1.70–2.20 m (2H, C$^{4'}$$H_2$), 2.56–2.80 m (4H, C$^{3'}$$H_2$ + C$^{5'}$$H_2$), 3.52 s (2H, C$^{1'}$$H_2$), 3.59 s (2H, C^{3}$H_2$), 3.81 s (3H, OCH_3), 4.57 s (2H, NC$\underline{H}_2$Ar), 6.70–8.00 m (10H_{arom}). Found, %: C 78.12; H 6.30; N 3.51. $C_{26}H_{25}NO_3$. Calculated, %: C 78.17; H 6.31; N 3.51.

4-(3,4-Dimethoxybenzyl)-1,4-dihydro-3*H*-spiro[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-dione (VIIb). Yield 88%, mp 207°C. IR spectrum, ν, cm^{-1}: 3000–3100 (C–H_{arom}), 2800–3000 (C–$H_{aliphat}$), 1715 (C=O), 1685 (C=O), 1261 (C–O–C), 1224 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 1.70–2.20 m (2H, C$^{4'}$$H_2$), 2.56–2.90 m (4H, C$^{3'}$$H_2$ + C$^{5'}$$H_2$), 3.52 s (2H, C$^{1'}$$H_2$), 3.59 s (2H, C^{3}$H_2$), 3.84, 3.90 s (3H, OCH_3), 4.56 s (2H, NC$\underline{H}_2$Ar), 6.80–8.00 m (9H_{arom}). Found, %: C 75.52; H 6.35; N 3.26. $C_{27}H_{27}NO_4$. Calculated, %: C 75.50; H 6.34; N 3.26.

4-(1,3-Benzodioxol-5-ylmethyl)-1,4-dihydro-3*H*-spiro[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-dione (VIIc). Yield 82%, mp 177°C. IR spectrum, ν, cm^{-1}: 3000–3100 (C–H_{arom}), 2800–3000 (C–$H_{aliphat}$), 1728 (C=O), 1699 (C=O), 1257 (C–O–C), 1239 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 1.70–2.30 m (2H, C$^{4'}$$H_2$), 2.60–2.90 m (4H, C$^{3'}$$H_2$ + C$^{5'}$$H_2$), 3.53 s (2H, C$^{1'}$$H_2$), 3.61 s (2H, C^{3}$H_2$), 4.55 s (2H, NC$\underline{H}_2$Ar), 5.96 s (2H, OCH_2O), 6.77–8.00 m (9H_{arom}). Found, %: C 75.54; H 5.61; N 3.40. $C_{26}H_{23}NO_4$. Calculated, %: C 75.53; H 5.61; N 3.39.

4-[(1,1'-Biphenyl)-4-ylmethyl]-1,4-dihydro-3*H*-spiro[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-dione (VII d). Yield 81%, mp 188°C. IR spectrum, ν, cm $^{-1}$: 3000–3100 (C–H_{arom}), 2800-3000 (C–$H_{aliphat}$), 1723 (C=O), 1700 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.68–2.31 m (2H, C$^{4'}$$H_2$), 2.56–2.91 m (4H, C$^{3'}$$H_2$+ C$^{5'}$$H_2$), 3.52 s (2H, C$^{1'}$$H_2$), 3.62 s (2H, C^{3}$H_2$), 4.68 s (2H, NC$\underline{H}_2$Ar), 6.90–8.00 m (15$H_{arom}$). Found, %: C 83.55; H 6.20; N 3.14. $C_{31}H_{27}NO_2$. Calculated, %: C 83.57; H 6.11; N 3.14.

4-(4-Isopropylbenzyl)-1,4-dihydro-3*H*-spiro-[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-dione (VII e). Yield 74%, mp 120°C. IR spectrum, ν, cm^{-1}: 3000–3100 (C–H_{arom}), 2953, 2920, 2872 (strong, C–$H_{aliphat}$), 1728 (C=O), 1698 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.24, 1.30 s [3H, CH(C$\underline{H}_3$)$_2$], 1.70–2.20 m (2H, C$^{4'}$$H_2$), 2.56–3.15 m [5H, C$^{3'}$$H_2$ + C$^{5'}$$H_2$ + C$\underline{H}$(CH_3)$_2$], 3.52 s (2H, C$^{1'}$$H_2$), 3.60 s (2H, C^{3}$H_2$), 4.57 s (2H, NC$\underline{H}_2$Ar), 6.90–8.00 m (10$H_{arom}$). Found, %: C 81.75; H 7.09; N 3.39. $C_{28}H_{29}NO_2$. Calculated, %: C 81.72; H 7.10; N 3.40.

4-Benzyl-4',4'-dimethyl-1,4-dihydro-3*H*-spiro[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-diones VIIf–VIIj were obtained analogously to compounds **VIIa– VIIe** from 0.001 mol of an appropriate amine **VIa–VIe**, 0.12 g (0.004 mol) of paraformaldehyde, and 0.14 g (0.001 mol) of dimedone (**IIb**).

4',4'-Dimethyl-4-(4-methoxybenzyl)-1,4-dihydro-3*H*-spiro[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-dione (VIIf). Yield 91%, mp 216°C. IR spectrum, ν, cm^{-1}: 3100–3000 (C–H_{arom}), 2960–2800 (C–$H_{aliphat}$), 1725 (C=O), 1697 (C=O), 1237 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 0.95, 1.10 s (3H, C$^{4'}$$CH_3$), 2.50, 2.70 d (2H, C$^{3'}$H + C$^{5'}$H, *J* 14 Hz), 3.50 s (2H, C$^{1'}$$H_2$), 3.56 s (2H, C^{3}$H_2$), 3.81 s (3H, OCH_3), 4.56 s (2H, NC$\underline{H}_2$Ar), 6.85–8.00 m (10H_{arom}). Found, %: C 78.63; H 6.87; N 3.27. $C_{28}H_{29}NO_3$. Calculated, %: C 78.66; H 6.84; N 3.28.

4-(3,4-Dimethoxybenzyl)-4',4'-dimethyl-1,4-dihydro-3*H*-spiro[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-dione (VIIg). Yield 93%, mp 217°C. IR spectrum, ν, cm^{-1}: 3100–3000 (C–H_{arom}), 3000–2800 (C–$H_{aliphat}$),

1716 (C=O), 1684 (C=O), 1260 (C–O–C), 1227 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 0.97, 1.09 s (3H, C$^{4'}$CH$_3$), 2.50, 2.70 d (2H, C$^{3'}$H + C$^{5'}$H, *J* 16 Hz), 3.50 s (2H, C$^{1'}$H$_2$), 3.56 s (2H, C^{3}H$_2$), 3.84, 3.90 s (3H, OCH$_3$), 4.56 s (2H, NCH$_2$Ar), 6.80–8.00 m (9H$_{arom}$). Found, %: C 76.12; H 6.82; N 3.07. $C_{29}H_{31}NO_4$. Calculated, %: C 76.12; H 6.83; N 3.06.

4-(1,3-Benzodioxol-5-ylmethyl)-4',4'-dimethyl-1,4-dihydro-3*H*-spiro[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-dione (VIIh). Yield 89%, mp 219°C IR spectrum, ν, cm^{-1}: 3100–3000 (C–H$_{arom}$), 3000–2800 (C–H$_{aliphat}$), 1727 (C=O), 1698 (C=O), 1256 (C–O–C), 1240 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 0.98, 1.11 s (3H, C$^{4'}$CH$_3$), 2.52, 2.72 d (2H, C$^{3'}$H + C$^{5'}$H, *J* 16 Hz), 3.50 s (2H, C$^{1'}$H$_2$), 3.57 s (2H, C^{3}H$_2$), 4.54 s (2H, NCH$_2$Ar), 5.96 s (2H, OCH$_2$O), 6.77–8.00 m (9H$_{arom}$). Found, %: C 76.20; H 6.17; N 3.12. $C_{28}H_{27}NO_4$. Calculated, %: C 76.17; H 6.16; N 3.17.

4-[(1,1'-Biphenyl)-4-ylmethyl]-4',4'-dimethyl-1,4-dihydro-3*H*-spiro[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-dione (VIIi). Yield 83%, mp 209°C. IR spectrum, ν, cm^{-1}: 3100–3000 (C–H$_{arom}$), 3000–2800 (C–H$_{aliphat}$), 1725 (C=O), 1700 (C=O). ^{1}H NMR spectrum, δ, ppm: 0.97, 1.08 s (3H, C$^{4'}$CH$_3$), 2.52, 2.73 d (2H, C$^{3'}$H + C$^{5'}$H, *J* 16 Hz), 3.52 s (2H, C$^{1'}$H$_2$), 3.63 s (2H, C^{3}H$_2$), 4.68 s (2H, NCH$_2$Ar), 6.90–8.00 m (15H$_{arom}$). Found, %: C 83.71; H 6.63; N 2.99. $C_{33}H_{31}NO_2$. Calculated, %: C 83.69; H 6.60; N 2.96.

4-(4-Isopropylbenzyl)-4',4'-dimethyl-1,4-dihydro-3*H*-spiro[benzo[*f*]quinoline-2,1'-cyclohexane]-2',6'-dione (VII j). Yield 82%, mp 192°C. IR spectrum, ν, cm^{-1}: 3100–3000 (C–H$_{arom}$), 2956, 2924, 2871 (CandльНые, C–H$_{aliphat}$), 1728 (C=O), 1697 (C=O). ^{1}H NMR spectrum, δ, ppm: 0.94, 1.08 s (3H, C$^{4'}$CH$_3$), 1.24, 1.30 s [3H, CH(CH$_3$)$_2$], 2.45, 2.65 d (2H, C$^{3'}$H + C$^{5'}$H, *J* 16 Hz), 2.90 m [1H, CH(CH$_3$)$_2$], 3.50 s (2H, C$^{1'}$H$_2$), 3.56 s (2H, C^{3}H$_2$), 4.58 s (2H, NCH$_2$Ar), 6.90–8.00 m (10H$_{arom}$). Found, %: C 81.99; H 7.57; N 3.12. $C_{30}H_{33}NO_2$. Calculated, %: C 81.97; H 7.57; N 3.19.

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