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Synthesis of 4-Alkyl(aryl, hetaryl)-2-thioxo-5,6,7,8-tetrahydroquinoline-3-carbonitriles and Their Derivatives by Cross-Recyclization of 4-Alkyl(aryl, hetaryl)-2,6-diamino-4*H*-thiopyran-3,5-dicarbonitriles with 4-(Cyclohex-1-en-1-yl)morpholine, Alkyl Halides, and Cyclohexanone

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Abstract—Cross recyclization of 4-substituted 2,6-diamino-4*H*-thiopyran-3,5-dicarbonitriles with 4-(cyclohex-1-en-1-yl)morpholine, alkyl halides, and cyclohexanone gave the corresponding substituted 2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitriles, 2-alkylsulfanyl-5,6,7,8-tetrahydroquinoline-3-carbonitriles, 3-amino-5,6,7,8-tetrahydrothieno[2,3-*b*]quinoline-2-carboxamides, and 4-oxo-2,2-pentamethylene-1,2,3,4,7,8,9,10-octahydropyrimido[4′,5′:4,5]thieno[2,3-*b*]quinolines. The structure of 3-(4-bromophenyl)-2,2-pentamethylene-11-(2-thienyl)-1,2,3,4,7,8,9,10-octahydropyrimido[4′,5′:4,5]thieno[2,3-*b*]quinoline was proved by X-ray analysis.

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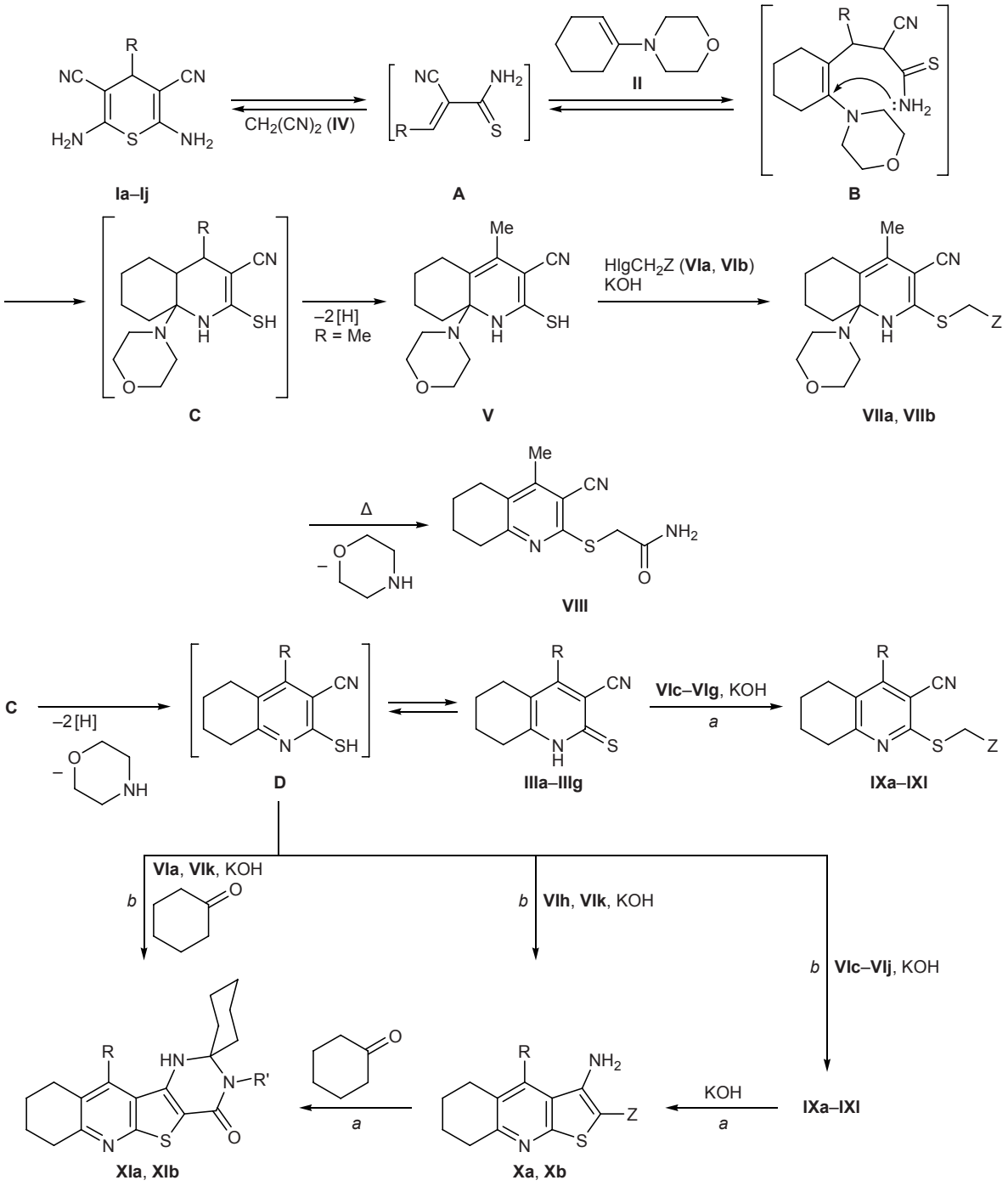
It was shown previously [1] that cross-recyclization of 4-aryl-2,6-diamino-4*H*-thiopyran-3,5-dicarbonitriles with hydrazines leads to the formation of 4-aryl-2,6-dihydrazinopyridine-3,5-dicarbonitriles [1]. Cross-recyclization of the same thiopyran derivatives with pyridinium ylides gives substituted 3,4-*trans*-4-aryl-5-cyano-3-pyridinio-1,2,3,4-tetrahydropyridine-6-thiolates [2], while analogous reactions with ketones and 1,3-diketones provide substituted 4-aryl-3-cyanopyridine-2(1*H*)-thiones and 2-amino-4-aryl-3-cyano-5,6,7,8-tetrahydro-4*H*-benzo[*b*]pyran-5-ones [3].

In the present article we report on a new cross-recyclization of 4-alkyl(aryl, hetaryl)-2,6-diamino-4*H*-thiopyran-3,5-dicarbonitriles **Ia**–**Ij** with 4-(cyclohex-1-en-1-yl)morpholine (**II**) in boiling anhydrous ethanol. The products were the corresponding 4-substituted 2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitriles **IIIa**–**IIIg** (Scheme 1). The same compounds were synthesized previously from aryl(hetaryl)methylidene(cyano)thioacetamides and cyclohexanone [4] or 4-(cyclohex-1-en-1-yl)morpholine [5], as well as by condensation of 2-arylmethylidenecyclohexan-1-ones with cyano(thioacetamide) [6] or recyclization of 4-amino-6-aryl-2,2-pentamethylene-1,3-dithiacyclohex-4-ene-5-carbonitriles [7].

Presumably, the examined cross-recyclization involves opening of the thiopyran ring with formation of malononitrile (**IV**) and R-methylidene(cyano)thioacetamide **A**. Stork alkylation of the latter with enamine **II** [8] gives intermediate **C** which undergoes heterocyclization to substituted octahydroquinoline **C**. Depending on the R substituent, quinoline derivatives **C** can be transformed along two pathways. The first of these is dehydrogenation to 4-methyl-2-sulfanyl-8a-morpholino-1,5,6,7,8,8a-hexahydroquinoline-3-carbonitrile (**V**), and the second involves simultaneous dehydrogenation and elimination of morpholine to form 4-substituted 2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitriles **IIIa**–**IIIg** (Scheme 1). The structure of compounds **IIIa**–**IIIg** and **V** was confirmed by their physical constants and spectral data (see Experimental), as well as by chemical tranformations. Alkylation of thiol **V** with chloroacetamide (**VIa**) and chloroacetonitrile (**VIb**) in DMF in the presence of potassium hydroxide as a base gave sulfides **VIIa** and **VIIb**, respectively. Elimination of morpholine molecule from compound **VIIa** on heating in glacial acetic acid resulted in the formation of tetrahydroquinoline **VIII**.

The alkylation of partially hydrogenated substituted quinolinethiones **IIIa**–**IIIg** with alkyl halides **VIc**–**VIg**

Scheme 1.



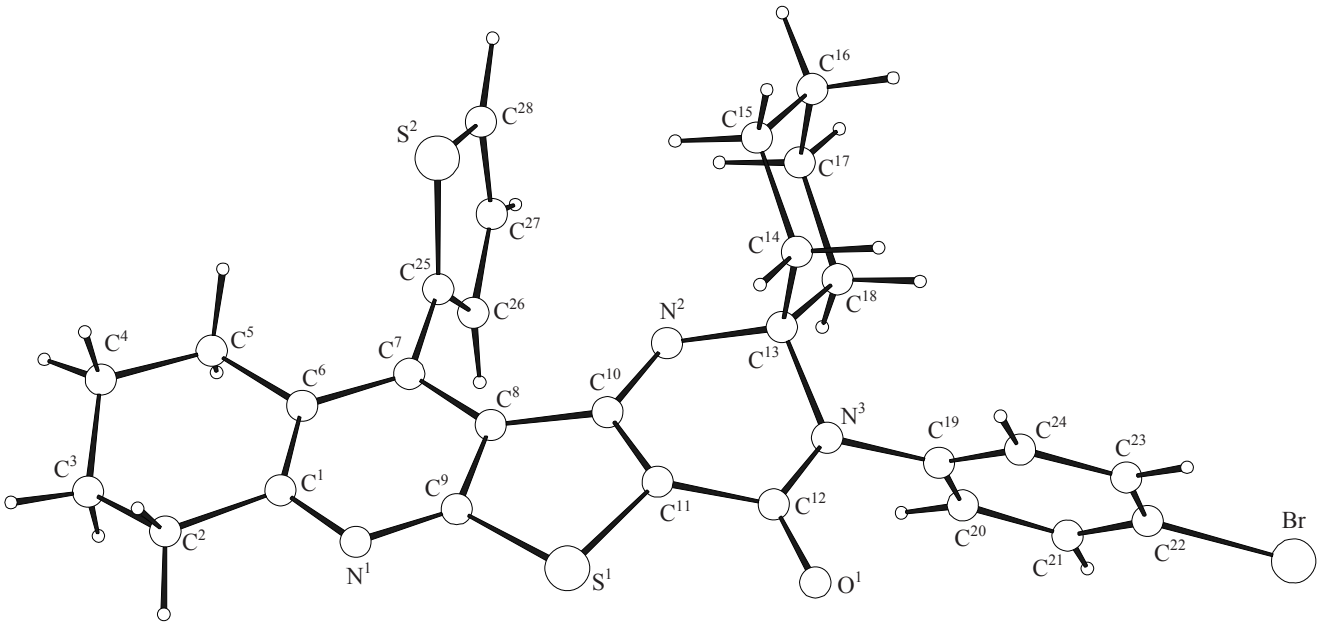
I, R = Me_2CHCH_2 (**a**), 2-MeC_6H_4 (**b**), 4-$O_2NC_6H_4$ (**c**), 4-BrC_6H_4 (**d**), Ph (**e**), 4-FC_6H_4 (**f**), 2-thienyl (**g**), Me (**h**), 5-methylfuran-2-yl (**i**), 4-$Me_2CHC_6H_4$ (**j**); **III**, R = 2-MeC_6H_4 (**a**), 4-FC_6H_4 (**b**), Ph (**c**), 4-BrC_6H_4 (**d**), Me_2CHCH_2 (**e**), 2-thienyl (**f**), 4-$O_2NC_6H_4$ (**g**); **VI**, Hlg = Cl (**a**, **b**, **h**, **i**, **k**), Br (**c**–**g**, **j**); Z = $CONH_2$ (**a**), CN (**b**), 2-oxo-2*H*-chromen-3-ylcarbonyl (**c**), 4-ClC_6H_4CO (**d**), naphthalen-2-ylcarbonyl (**e**), CH_2=CH (**f**), 4-BrC_6H_4CO (**g**), PhNHCO (**h**), 3,4-$(HO)_2C_6H_3CO$ (**i**), EtOCO (**j**), 4-BrC_6H_4NHCO (**k**); **VII**, Z = $CONH_2$ (**a**), CN (**b**); **IX**, R = 4-FC_6H_4, Z = 4-ClC_6H_4CO (**a**), R = 4-FC_6H_4, Z = 2-oxo-2*H*-chromen-3-ylcarbonyl (**b**), R = Me_2CHCH_2, Z = naphthalen-2-ylcarbonyl (**c**), R = 4-$O_2NC_6H_4$, Z = 2-oxo-2*H*-chromen-3-ylcarbonyl (**d**); R = 4-BrC_6H_4, Z = 4-BrC_6H_4CO (**e**); R = 4-BrC_6H_4, Z = CH_2=CH (**f**); R = Ph, Z = 4-ClC_6H_4CO (**g**); R = 4-$Me_2CHC_6H_4$, Z = 2-oxo-2*H*-chromen-3-ylcarbonyl (**h**); R = 4-$Me_2CHC_6H_4$, Z = PhNHCO (**i**); R = 5-methylfuran-2-yl, Z = EtOCO (**j**); R = 5-methylfuran-2-yl, Z = 4-ClC_6H_4CO (**k**); R = 5-methylfuran-2-yl, Z = 3,4-$(HO)_2C_6H_3CO$ (**l**); **X**, R = 4-$Me_2CHC_6H_4$, Z = PhNHCO (**a**); R = 2-thienyl, Z = 4-BrC_6H_4NHCO (**b**); **XI**, R = 2-thienyl, R′ = 4-BrC_6H_4 (**a**), R = Me_2CHCH_2, R′ = H (**b**).

under analogous conditions regioselectively afforded sulfides **IXa**–**IXl** (method *a*). Compounds **IX** can also be synthesized in a one-pot mode from the corresponding thiopyrans **I**, enamine **II**, and alkyl halides **VI** without intermediate isolation of tetrahydroquinolines **III** (method *b*).

Sulfides **IX** are convenient as starting compounds for the synthesis of fused systems containing a tetrahydroquinoline fragment, e.g., 5,6,7,8-tetrahydrothieno-[2,3-*b*]quinoline [9] and 1,2,3,4,7,8,9,10-octahydropyrimido[4′,5′:4,5]thieno[2,3-*b*]quinoline [10]. Treatment of substituted 2-(phenylcarbamoylmethylsulfanyl)-5,6,7,8-tetrahydroquinoline **IXi** with alkali gave 3-amino-2-phenylcarbamoyl-5,6,7,8-tetrahydrothieno-[2,3-*b*]quinoline **Xa** (method *a*) which attracts interest as intermediate product for the synthesis of compounds possessing antimicrobial activity [11]. Compounds **Xa** and **Xb** are also readily obtained by cross-recyclization of thiopyrans **Ia** and **Ig** with morpholinocyclohexene **II** and alkyl halides **VIc** and **VIj**, followed by treatment of the reaction mixture with alkali (method *b*). Presumably, in this case intermediate thiol **D** undergoes alkylation to sulfide **IX**, and the subsequent intramolecular cyclization of **IX** yields substituted thienoquinolines **X**.

The structure of compounds **IX** and **X** is confirmed by spectral data. Sulfides **IX** characteristically displayed in the IR spectra absorption band at 2218–2225 cm^{-1} due to stretching vibrations of the conjugated cyano group. No such band was present in the IR spectra of 3-aminothienoquinolines **X**; instead, stretching and bending vibrations of the amino group in **X** gave rise to absorption bands at 3190–3480 and 1645–1666 cm^{-1}, respectively. The ^1H NMR spectra of **IX** contained signals from protons in the tetrahydroquinoline fragment and substituents therein (see Experimental) and singlets from the SCH_2 protons at δ 4.01–4.89 ppm. The latter signal was lacking in the spectra of compounds **X**, but broadened singlets from protons in the amino group appeared in the region δ 5.64–6.13 ppm; these data are consistent with the cyclization of **IX** into substituted partially hydrogenated thienoquinolines **X**. Apart from spectral methods (see Experimental), the structure of compounds **X** was proved by chemical transformations. The known condensation of cyclohexanone with heterocyclic compounds containing amino and carboxamide groups at neighboring carbon atoms [10, 12] gave the corresponding spiro-fused heterocyclic systems **XIa** and **XIb** (method *a*) which are potential antimicrobial agents [12]. The same compounds were synthesized by one-pot reaction of thiopyrans **I** with enamine **II**, alkyl halides **VIa** and **VIk**, and cyclohexanone (method *b*).

The structure of 3-(4-bromophenyl)-2,2-pentamethylene-11-(2-thienyl)-1,2,3,4,7,8,9,10-octahydropyrimido[4′,5′:4,5]thieno[2,3-*b*]quinoline (**XIa**) was un-



Structure of the molecule of 3-(4-bromophenyl)-2,2-pentamethylene-11-(2-thienyl)-1,2,3,4,7,8,9,10-octahydropyrimido[4′,5′:4,5]-thieno[2,3-*b*]quinolin-4-one (**XIa**) according to the X-ray diffraction data. Principal bond lengths (Å) and bond angles (deg): S^1–C^9 1.77(1), S^1–C^{11} 1.76(1), N^1–C^1 1.34(1), N^1–C^9 1.32(1), N^2–C^{10} 1.37(1), N^2–C^{13} 1.48(1), N^3–C^{12} 1.33(1), N^3–C^{13} 1.51(1), $C^9S^1C^{11}$ 90.2(5), $C^1N^1C^9$ 115.4(9), $C^{10}N^2C^{13}$ 117.6(7), $C^{12}N^3C1^{13}$ 120.2(7).

ambiguously proved by X-ray analysis (see figure). We thus confirmed the direction of cross-recyclization of thiopyrans **I** with enamine **II**. The central bicyclic fragment $S^1N^1C^1C^6$–C^{11} in molecule **XIa** is planar within 0.025 Å; the dihedral angle between the $N^1C^1C^6$–C^9 and $S^1C^1C^9$–C^{11} rings is as small as 1.3°. The bond length distribution in the bicyclic system suggests essential electron density delocalization. The S^2C^{25}–C^{28} thiophene ring is almost orthogonal to the above plane for steric reasons: the corresponding dihedral angle is equal to 86.0°. The six-membered ring $N^2N^3C^{11}$–C^{13} is appreciably nonplanar: deviations of atoms therein from the mean-square plane reach 0.294 Å; it adopts a *half-chair* conformation with the following Cramer–Pople parameters [13]: $S = 0.63$, $\Psi = 7.6$, and $\theta = 53.1°$. The benzene ring C^{19}–C^{24} forms with the $N^2N^3C^{11}$–C^{13} ring a dihedral angle of 74.9°. The cyclohexane ring C^{13}–C^{18} has an almost undistorted *chair* conformation which is typical of cyclohexane derivatives: the endocyclic angles vary within a narrow range, from 53.5 to 55.7°. On the other hand, the C^1–C^6 cyclohexane ring fused to the bicyclic system $S^1N^1C^1C^6$–C^{11} exists in a *half-boat* conformation ($S = 0.69$, $\Psi = 28.7$, $\theta = 37.6°$). The N^3 atom has a planar–trigonal bond configuration [the sum of the bond angles is 358(2)°]. Effective conjugation between the lone electron pair on N^3 and π system of the $C^1{=}O^1$ double bond induces shortening of the N^3–C^{12} bond to 1.37(1) Å against the standard purely single $N(sp^2)$–$C(sp^2)$ bond (1.43–1.45 Å [14, 15]).

EXPERIMENTAL

The X-ray diffraction data for a 0.37-mm spherical single crystal of **XIa** were acquired at room temperature on an Enraf–Nonius CAD-4 automatic four-circle diffractometer (MoK_α irradiation, $\lambda = 0.71169$ Å, scan rate ratio $2\theta/\omega = 1.2$, $\theta_{max} = 27°$, spherical segment $0 \le h \le 13$, $0 \le k \le 30$, $-12 \le l \le 12$). Total of 4916 reflections were measured, 4488 of which were symmetry-independent ($R_{int} = 0.062$). Monoclinic crystals with the following unit cell parameters: $a = 10.418(2)$, $b = 23.859(3)$, $c = 10.060(4)$ Å; $\beta = 92.56(2)°$; $V = 2948(2)$ Å^3; $Z = 4$; $d_{calc} = 1.50$ g/cm^3; $\mu = 18.1$ cm^{-1}; $F(000) = 1155.6$, space group $P2_1/c$ (no. 14). The structure was solved by the direct method and was refined by the least-squares procedure in full-matrix anisotropic approximation using CRYSTALS software package [16]. The refinement procedure involved 1593 reflections with $I > 2\sigma(I)$ (316 refined parameters, 5.1 reflections per parameter). The positions of 90% of hydrogen atoms were determined by difference synthesis of electron density, and the positions of the other hydrogen atoms were calculated from geometry considerations. All hydrogen atoms were included in the refinement procedure with fixed thermal and positional parameters. Chebyshev's weight scheme [17] with the following four parameters was used: 1.22, –0.91, 0.73, –0.39. The final divergence factors were $R = 0.061$ and $R_W = 0.061$, goodness of fit 1.171. The residual electron density from the Fourier difference series was 0.55 and –0.59 e/Å^3. Absorption by the crystal was taken into account by the azimuthal scanning technique [18].

The IR spectra were recorded in mineral oil on an IKS-40 spectrometer. The ^{1}H NMR spectra were measured on Bruker WP-100SY (100 MHz; **V**, **VIIa**, **VIIb**, **VIII**, **IXa**, **IXb**, **IXf**, **IXg**, **IXk**), Gemini-200 (199.975 MHz; **IIIa**–**IIIe**, **XIa**), Bruker AC-200 (200.13 MHz; **IXi**), Bruker WM-250 (250.13 MHz; **IXc**), Bruker AM-300 (300.13 MHz; **IXj**, **IXl**), Varian Mercury-400 (400.397 MHz; **Ii**, **Ij**, **IXh**, **XIb**), and Bruker DRX 500 instruments (500.13 MHz; **IXd**, **IXe**, **Xa**, **Xb**) using DMSO-d_6 as solvent and tetramethylsilane as internal reference. The mass spectra (electron impact, 70 eV) were obtained on a Kratos MS-890 mass spectrometer with direct sample admission into the ion source (**IXe**, **IXj**, **Xa**, **Xb**) and a Hewlett–Packard Chrommas 5890/5972 GC–MS system (**IIIf**, **XIb**; HP-5 MS column; samples were injected as solutions in methylene chloride). The melting points were determined on a Kofler hot stage. The progress of reactions and the purity of products were monitored by TLC on Silufol UV-254 plates using acetone–hexane (3 : 5) as eluent; spots were visualized by treatment with iodine vapor and UV irradiation.

4-Substituted 2,6-diamino-4*H*-thiopyran-3,5-dicarbonitriles **Ia**–**Ij** were synthesized according to the procedure described in [19]. The properties of compounds **Ia** [20], **Ib** [21], **Ic** [22], **Id**–**If** [19], **Ig** [23], and **Ih** [24] were reported previously.

2,6-Diamino-4-(5-methylfuran-2-yl)-4*H*-thiopyran-3,5-dicarbonitrile (Ii). Yield 1.83 g (71%), colorless wool-like material, mp 182–184°C (from AcOH). IR spectrum, ν, cm^{-1}: 3212, 3333, 3479 (NH_2); 2210 sh (C≡N); 1647 (δNH_2). ^{1}H NMR spectrum, δ, ppm: 2.26 s (3H, Me), 4.18 s (1H, 4-H), 6.89 d (1H, 3′-H, $J = 2.95$ Hz), 6.98 d (1H, 4′-H), 6.67 br.s (4H, NH_2). Found, %: C 55.71; H 3.77; N 21.50. $C_{12}H_{10}N_4OS$. Calculated, %: C 55.80; H 3.90; N 21.69.

2,6-Diamino-4-(4-isopropylphenyl)-4*H*-thiopyran-3,5-dicarbonitrile (Ij). Yield 2.40 g (81%), yellow

crystals, mp 236–239°C (from EtOH). IR spectrum, ν, cm^{-1}: 3190, 3327, 3465 (NH_2); 2200 sh (C≡N); 1650 (δNH_2). ^{1}H NMR spectrum, δ, ppm: 1.24 d (6H, Me, J = 7.12 Hz), 2.86 m (1H, C**H**Me_2), 4.15 s (1H, 4-H), 6.54 br.s (4H, NH_2), 7.16 d and 7.22 d (2H each, C_6H_4, J = 7.13 Hz). Found, %: C 64.69; H 5.28; N 18.75. $C_{16}H_{16}N_4S$. Calculated, %: C 64.84; H 5.44; N 18.90. M 296.39.

4-Substituted 2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitriles IIIa–IIIg (*general procedure*). 4-(Cyclohex-1-en-1-yl)morpholine (**II**), 1.64 ml (10 mmol), was added to a suspension of 10 mmol of the corresponding thiopyran **I** in 25 ml of anhydrous ethanol, and the mixture was heated for 2 h under reflux. The mixture was cooled, diluted with 10% hydrochloric acid to pH 5, and left to stand for 24 h. The precipitate was filtered off and washed with water, ethanol, and hexane.

4-(2-Methylphenyl)-2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile (IIIa). Yield 1.79 g (64%), yellow powder, mp 226–228°C (from AcOH). IR spectrum, ν, cm^{-1}: 3335 (NH), 2225 (C≡N). ^{1}H NMR spectrum, δ, ppm: 1.65 m (4H, CH_2), 1.80 m (2H, CH_2), 2.12 s (3H, Me), 2.79 t (2H, CH_2, J = 6.18 Hz), 7.04 d (1H, H_{arom}, J = 6.14 Hz), 7.30 m (3H, H_{arom}), 13.86 br.s (1H, NH). Found, %: C 72.71; H 5.60; N 10.11. $C_{17}H_{16}N_2S$. Calculated, %: C 72.82; H 5.75; N 9.99.

4-(4-Fluorophenyl)-2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile (IIIb). Yield 1.96 g (69%), yellow crystals, mp 256–258°C (from AcOH); published data [25]: mp 258–259°C.

4-Phenyl-2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile (IIIc). Yield 1.86 g (70%), yellow powder, mp 240–242°C (from AcOH); published data [6]: mp 238°C (decomp.).

4-(4-Bromophenyl)-2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile (IIId). Yield 2.35 g (68%), yellow crystals, mp 260–262°C (from AcOH); published data [26]: mp 261–263°C.

4-Isobutyl-2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile (IIIe). Yield 1.48 g (60%), yellow powder, mp 199–201°C (from AcOH); published data [27]: mp 198–200°C.

4-(2-Thienyl)-2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile (IIIf). Yield 1.96 g (72%), yellow crystals, mp 215–217°C (from AcOH); published data [4]: mp 220–222°C. Mass spectrum: m/z 273 (I_{rel} = 100%) $[M + 1]^+$.

4-(4-Nitrophenyl)-2-thioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile (IIIg). Yield 2.46 g (79%), yellow powder, mp 252–254°C (from AcOH); published data [4]: mp 250°C (decomp.).

4-Methyl-8a-morpholino-2-sulfanyl-1,5,6,7,8,8a-hexahydroquinoline-3-carbonitrile (V). 4-(Cyclohex-1-en-1-yl)morpholine (**II**), 1.64 ml (10 mmol), was added to a suspension of 1.92 g (10 mmol) of thiopyran **Ih** in 25 ml of anhydrous ethanol, and the mixture was heated for 2 h under reflux. The mixture was cooled, and the precipitate was filtered off and washed with anhydrous ethanol and hexane. Yield 2.2 g (75%), yellow powder, mp 237–239°C (sublimes at 210°C). IR spectrum, ν, cm^{-1}: 3366 (NH), 2223 (C≡N). ^{1}H NMR spectrum, δ, ppm: 1.70 m (4H, CH_2), 2.30 s (3H, Me), 2.44 t (2H, CH_2, J = 7.12 Hz), 2.68 t (2H, CH_2, J = 7.41 Hz), 3.03 t (4H, CH_2NCH_2, J = 4.28 Hz), 3.70 t (4H, CH_2OCH_2), 8.43 br.s (2H, NH, SH). Found, %: C 61.72; H 7.19; N 14.25. $C_{15}H_{21}N_3OS$. Calculated, %: C 61.82; H 7.26; N 14.42. M 291.42.

(3-Cyano-4-methyl-8a-morpholino-1,5,6,7,8,8a-hexahydroquinolin-2-ylsulfanyl)acetamide (VIIa). Hexahydroquinoline **V**, 2.91 g (10 mmol), was dissolved in 10 ml of dimethylformamide, 5.6 ml (10 mmol) of 10% aqueous potassium hydroxide and 0.94 g (10 mmol) of chloroacetamide (**VIa**) were added in succession, and the mixture was stirred for 2 h and diluted with an equal volume of water. The precipitate was filtered off and washed with water, ethanol, and hexane. Yield 2.92 g (84%), white powder, mp 175–177°C. IR spectrum, ν, cm^{-1}: 3159, 3311, 3402, 3478 (NH, NH_2); 2228 (C≡N); 1690 (CONH). ^{1}H NMR spectrum, δ, ppm: 1.74 m (4H, CH_2), 2.32 s (3H, Me), 2.44 m (2H, CH_2), 2.79 m (2H, CH_2), 3.04 m (4H, CH_2OCH_2), 3.86 m (6H, SCH_2, CH_2NCH_2), 7.12 br.s and 7.49 br.s (1H each, NH_2), 7.87 br.s (1H, NH). Found, %: C 58.49; H 7.09; N 15.88. $C_{17}H_{24}N_4O_2S$. Calculated, %: C 58.60; H 6.94; N 16.08. M 348.47.

2-(Cyanomethylsulfanyl)-4-methyl-8a-morpholino-1,5,6,7,8,8a-hexahydroquinoline-3-carbonitrile (VIIb) was synthesized in a similar way from 0.63 ml (10 mmol) of chloroacetonitrile (**VIb**). Yield 2.61 g (79%), white powder, mp 161–162°C. IR spectrum, ν, cm^{-1}: 3321 (NH); 2258, 2222 (C≡N). ^{1}H NMR spectrum, δ, ppm: 1.78 m (4H, CH_2), 2.36 s (3H, Me), 2.44 m (2H, CH_2), 2.88 m (6H, CH_2, CH_2NCH_2), 3.02 m (4H, CH_2OCH_2), 4.29 s (2H, SCH_2), 8.08 br.s (1H, NH). Found, %: C 61.65; H 6.62; N 17.09. $C_{17}H_{22}N_4OS$. Calculated, %: C 61.79; H 6.71; N 16.96.

(3-Cyano-4-methyl-5,6,7,8-tetrahydroquinolin-2-ylsulfanyl)acetamide (VIII) was obtained by recrystallization of 3.48 g (10 mmol) of compound **VIIa** from 15 ml of glacial acetic acid. Yield 2.01 g (77%), colorless crystals, mp 181–184°C. IR spectrum, ν, cm^{-1}: 3302, 3407, 3479 (NH_2); 2228 (C≡N); 1684 (CONH). ^{1}H NMR spectrum, δ, ppm: 1.78 m (4H, CH_2), 2.32 s (3H, Me), 2.59 m (2H, CH_2), 2.81 m (2H, CH_2), 3.88 s (2H, SCH_2), 7.12 br.s and 7.53 br.s (1H each, NH_2). Found, %: C 59.60; H 5.66; N 15.88. $C_{13}H_{15}N_3OS$. Calculated, %: C 59.75; H 5.79; N 16.08.

Compounds **IXa**–**IXl** were synthesized (method *a*) as described above for sulfide **VIIa** from the corresponding thioxoquinolines **IIIa**–**IIIg** and halogen derivatives **VIc**–**VIj**.

2-(4-Chlorobenzoylmethylsulfanyl)-4-(4-fluorophenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (IXa). Yield 3.22 g (74%), mp 222–224°C (from AcOH). IR spectrum, ν, cm^{-1}: 2220 (C≡N), 1710 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.45–1.84 m (4H, CH_2), 2.30 t (2H, CH_2, J = 17.12 Hz), 2.51 m (2H, CH_2), 4.81 s (2H, SCH_2), 7.36 m (4H, 4-FC_6H_4), 7.65 d and 8.11 d (2H each, 4-ClC_6H_4, J = 7.92 Hz). Found, %: C 65.81; H 4.01; N 6.29. $C_{24}H_{18}ClFN_2OS$. Calculated, %: C 65.98; H 4.15; N 6.41. M 436.94.

4-(4-Fluorophenyl)-2-(2-oxo-2*H*-chromen-3-ylcarbonylmethylsulfanyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (IXb). Yield 3.76 g (80%), mp 245–247°C (from AcOH). IR spectrum, ν, cm^{-1}: 2224 (C≡N); 1727, 1714 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.64 m (4H, CH_2), 2.30 t (2H, CH_2, J = 7.12 Hz), 2.67 t (2H, CH_2, J = 7.25 Hz), 4.79 s (2H, SCH_2), 7.43–7.51 m (6H, H_{arom}), 7.78 m (1H, H_{arom}), 7.98 d (1H, H_{arom}, J = 8.01 Hz), 8.76 s (1H, 4′-H, chromene). Found, %: C 68.84; H 3.95; N 5.81. $C_{27}H_{19}FN_2O_3S$. Calculated, %: C 68.92; H 4.07; N 5.95.

4-Isobutyl-2-(naphthalen-2-ylcarbonylmethylsulfanyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (IXc). Yield 3.27 g (79%), mp 154–156°C (from AcOH). IR spectrum, ν, cm^{-1}: 2225 (C≡N), 1709 (C=O). ^{1}H NMR spectrum, δ, ppm: 0.95 d (6H, Me, J = 5.13 Hz), 1.60–1.77 m (6H, CH_2), 1.95 m (1H, C**H**Me_2), 2.54 m (2H, CH_2), 2.66 d (2H, CH_2, J = 5.16 Hz), 4.87 s (2H, SCH_2), 7.51–7.73 m (2H, H_{arom}), 7.90–8.05 m (3H, H_{arom}), 8.11 d (1H, H_{arom}, J = 6.02 Hz), 8.76 s (1H, 1′-H). Found, %: C 75.20; H 6.19; N 6.58. $C_{26}H_{26}N_2OS$. Calculated, %: C 75.33; H 6.32; N 6.76.

4-(4-Nitrophenyl)-2-(2-oxo-2*H*-chromen-3-ylcarbonylmethylsulfanyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (IXd). Yield 3.88 g (78%), mp 259–261°C (from BuOH). IR spectrum, ν, cm^{-1}: 2219 (C≡N); 1730, 1712 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.59–1.84 m (4H, CH_2), 2.33 m (2H, CH_2), 2.79 m (2H, CH_2), 4.76 s (2H, SCH_2), 7.41 m (2H, H_{arom}), 7.61 d and 8.39 d (2H each, C_6H_4, J = 7.19 Hz), 7.75 m (1H, H_{arom}), 7.99 m (1H, H_{arom}), 8.73 s (1H, 4′-H, chromene). Found, %: C 65.00; H 3.72; N 8.33. $C_{27}H_{19}N_3O_5S$. Calculated, %: C 65.19; H 3.85; N 8.45.

2-(4-Bromobenzoylmethylsulfanyl)-4-(4-bromophenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (IXe). Yield 4.39 g (81%), mp 231–233°C (from AcOH). IR spectrum, ν, cm^{-1}: 2226 (C≡N), 1724 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.33–1.82 m (4H, CH_2), 2.25 m (2H, CH_2), 2.54 m (2H, CH_2), 4.86 s (2H, SCH_2), 7.31 d and 7.72 d (2H each, C_6H_4, J = 7.79 Hz), 7.87 d and 8.18 d (2H each, C_6H_4CO, J = 8.02 Hz). Found, %: C 52.98; H 3.21; N 4.95. $C_{24}H_{18}Br_2N_2OS$. Calculated, %: C 53.16; H 3.35; N 5.17.

2-Allylsulfanyl-4-(4-bromophenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (IXf). Yield 2.54 g (66%), mp 172–174°C (from MeOH). IR spectrum, ν, cm^{-1}: 2228 (C≡N). ^{1}H NMR spectrum, δ, ppm: 1.42–1.95 m (4H, CH_2), 2.34 t (2H, CH_2, J = 6.82 Hz), 2.94 t (2H, CH_2, J = 7.02 Hz), 3.94 d (2H, SCH_2, J = 6.58 Hz), 5.12 d (1H, =CH_2, J_{cis} = 9.55 Hz), 5.32 d (1H, =CH_2, J_{trans} = 17.40 Hz), 5.79–6.14 m (1H, CH=), 7.34 d and 7.74 d (2H each, H_{arom}, J = 7.25 Hz). Found, %: C 59.06; H 4.22; N 7.09. $C_{19}H_{17}Br_2N_2S$. Calculated, %: C 59.23; H 4.45; N 7.27.

2-(4-Chlorobenzoylmethylsulfanyl)-4-phenyl-5,6,7,8-tetrahydroquinoline-3-carbonitrile (IXg). Yield 3.01 g (72%), mp 198–200°C (from AcOH). IR spectrum, ν, cm^{-1}: 2226 (C≡N), 1727 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.48–1.82 m (4H, CH_2), 2.30 m (2H, CH_2), 2.85 m (2H, CH_2), 4.81 s (2H, SCH_2), 7.30–7.58 m (5H, Ph), 7.65 d and 8.11 d (2H each, C_6H_4, J = 7.99 Hz). Found, %: C 68.69; H 4.42; N 6.48. $C_{24}H_{19}ClN_2OS$. Calculated, %: C 68.81; H 4.57; N 6.69.

b. Enamine **II**, 1.64 ml (10 mmol), was added to a suspension of 10 mmol of the corresponding thiopyran **Ia**–**Ij** in 25 ml of anhydrous ethanol, and the mixture was heated for 2 h under reflux. The mixture was cooled, 5.6 ml (10 mmol) of 10% aqueous potassium hydroxide and 10 mmol of the corresponding halogen derivative **VIc**–**VIg** were added in succession under stirring, and the mixture was stirred for 4 h and diluted with an equal volume of water. The precipitate was filtered off, washed with water, ethanol, and hex-

ane, and recrystallized from glacial acetic acid. We thus obtained compounds **IXa**–**IXl**; among these, sulfides **IXa**–**IXg** were identical in the IR spectra, TLC data (R_f values), and melting points (no depression of the melting point was observed on mixing) to those synthesized according to method *a*. Yield, %: **IXa**, 74; **IXb**, 68; **IXc**, 80; **IXd**, 78; **IXe**, 72; **IXf**, 64; **IXg**, 58.

4-(4-Isopropylphenyl)-2-(2-oxo-2*H*-chromen-3-ylcarbonylmethylsulfanyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (IXh). Yield 3.80 g (77%), mp 240–242°C (from AcOH). IR spectrum, ν, cm^{-1}: 2222 (C≡N), 1717 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.26 d (6H, Me, J = 6.18 Hz), 1.61 m (2H, CH_2), 1.70 m (2H, CH_2), 2.35 t (2H, CH_2, J = 6.28 Hz), 2.71 t (2H, CH_2, J = 6.47 Hz), 2.99 m (1H, C**H**Me_2), 4.74 s (2H, SCH_2), 7.21 d and 7.38 d (2H each, C_6H_4, J = 7.02 Hz), 7.42 t (1H, H_{arom}, J = 7.62 Hz), 7.49 d (1H, H_{arom}, J = 7.58 Hz), 7.77 t (1H, H_{arom}, J = 7.62 Hz), 7.96 d (1H, H_{arom}, J = 7.85 Hz), 8.74 s (1H, 4′-H, chromene). Found, %: C 72.70; H 5.11; N 5.42. $C_{30}H_{26}N_2O_3S$. Calculated, %: C 72.85; H 5.30; N 5.66.

2-[3-Cyano-4-(4-isopropylphenyl)-5,6,7,8-tetrahydroquinolin-2-ylsulfanyl]-*N*-phenylacetamide (IXi). Yield 3.13 g (71%), mp 192–194°C (from AcOH). IR spectrum, ν, cm^{-1}: 2224 (C≡N), 1672 (CONH). ^{1}H NMR spectrum, δ, ppm: 1.29 d (6H, Me, J = 6.14 Hz), 1.52 m (2H, CH_2), 1.79 m (2H, CH_2), 2.38 t (2H, CH_2, J = 6.27 Hz), 2.88 t (2H, CH_2, J = 6.33 Hz), 2.99 m (1H, C**H**Me_2), 4.13 s (2H, SCH_2), 7.04 t (1H, C_6H_5, J = 6.95 Hz), 7.21 d and 7.39 d (2H each, C_6H_4, J = 7.06 Hz), 7.28 t (2H, C_6H_5, J = 6.94 Hz), 7.58 d (2H, C_6H_5, J = 6.96 Hz), 10.14 br.s (1H, NH). Mass spectrum, m/z (I_{rel}, %): 442 (4) $[M + 1]^+$, 441 (9) $[M]^+$, 440 (5) $[M - 1]^+$, 349 (100) $[M - PhNH]^+$, 321 (72), 279 (84), 265 (10), 205 (11), 132 (8), 93 (32) $[PhNH_2]^+$, 77 (21) $[Ph]^+$, 65 (14), 43 (12) $[CHMe_2]^+$. Found, %: C 73.28; H 5.95; N 9.38. $C_{27}H_{27}N_3OS$. Calculated, %: C 73.44; H 6.16; N 9.52. M 441.60.

Ethyl [3-cyano-4-(5-methylfuran-2-yl)-5,6,7,8-tetrahydroquinolin-2-ylsulfanyl]acetate (IXj). Yield 2.88 g (81%), mp 92–93°C (from EtOH). IR spectrum, ν, cm^{-1}: 2214 (C≡N), 1740 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.20 t (3H, $CH_2C\mathbf{H}_3$, J = 6.19 Hz), 1.68 m (2H, CH_2), 1.77 m (2H, CH_2), 2.36 s (3H, Me), 2.68 t (2H, CH_2, J = 6.22 Hz), 2.84 t (2H, CH_2, J = 6.19 Hz), 4.01 s (2H, SCH_2), 4.12 q (2H, OCH_2, J = 6.19 Hz), 6.38 d (1H, 3′-H, J = 2.14 Hz), 6.93 d (1H, 4′-H, J = 2.14 Hz). Found, %: C 63.89; H 5.42; N 7.76. $C_{19}H_{20}N_2O_3S$. Calculated, %: C 64.02; H 5.66; N 7.86.

2-(4-Chlorobenzoylmethylsulfanyl)-4-(5-methylfuran-2-yl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (IXk). Yield 3.59 g (85%), mp 150–151°C (from AcOH). IR spectrum, ν, cm^{-1}: 2216 (C≡N), 1710 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.61–1.84 m (4H, CH_2), 2.42 s (3H, Me), 2.59 m (2H, CH_2), 2.24 m (2H, CH_2), 4.69 s (2H, SCH_2), 6.30 d (1H, 3′-H, J = 2.15 Hz), 6.87 d (1H, 4′-H, J = 2.15 Hz), 7.52 d and 8.07 d (2H each, C_6H_4, J = 8.57 Hz). Found, %: C 65.13; H 4.39; N 6.52. $C_{23}H_{19}ClN_2O_2S$. Calculated, %: C 65.32; H 4.53; N 6.62.

2-(3,4-Dihydroxybenzoylmethylsulfanyl)-4-(5-methylfuran-2-yl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (IXl). Yield 3.36 g (80%), white powder, mp 171–173°C (from AcOH). IR spectrum, ν, cm^{-1}: 3540 (OH), 2218 (C≡N), 1704 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.61–1.89 m (4H, CH_2), 2.40 s (3H, Me), 2.62–2.81 m (4H, CH_2), 4.68 s (2H, SCH_2), 6.32 d (1H, 3′-H, J = 2.84 Hz), 6.80–6.92 m (2H, H_{arom}), 7.35–7.49 m (2H, H_{arom}), 9.14 br.s and 9.62 br.s (1H each, OH). Mass spectrum, m/z (I_{rel}, %): $[M]^+$ is missing, 283 (43) $[M - 3,4\text{-}(HO)_2C_6H_3CO]^+$, 270 (35), 269 (14), 241 (22), 227 (20), 209 (11), 200 (18), 152 (44), 137 (100) $[3,4\text{-}(HO)_2C_6H_3CO]^+$, 109 (33), 81 (22), 77 (17) $[Ph]^+$, 65 (11), 63 (23). Found, %: C 65.52; H 4.61; N 6.49. $C_{23}H_{20}N_2O_4S$. Calculated, %: C 65.70; H 4.79; N 6.66. M 420.49.

3-Amino-4-(4-isopropylphenyl)-*N*-phenyl-5,6,7,8-tetrahydrothieno[2,3-*b*]quinoline-2-carboxamide (Xa). *a*. Compound **IXi**, 4.41 g (10 mmol), was dissolved in 10 ml of DMF, 5.6 ml (10 mmol) of 10% aqueous potassium hydroxide was added, and the mixture was stirred for 5 h and diluted with an equal volume of water. The precipitate was filtered off and washed in succession with water, ethanol, and hexane. Yield 3.66 g (83%), yellow crystals, mp 235–237°C (from AcOH). IR spectrum, ν, cm^{-1}: 3195, 3288, 3410 (NH_2); 1674 (CONH), 1641 (δNH_2). ^{1}H NMR spectrum, δ, ppm: 1.31 d (6H, Me, J = 6.13 Hz), 1.72 m (2H, CH_2), 1.89 m (2H, CH_2), 2.40 t (2H, CH_2, J = 6.31 Hz), 3.02 m (3H, CH_2, C**H**Me_2), 5.64 br.s (2H, NH_2), 7.02 t (1H, H_{arom}, J = 6.92 Hz), 7.28 m (4H, H_{arom}), 7.45 d (2H, H_{arom}, J = 7.22 Hz), 7.63 d (2H, H_{arom}, J = 7.34 Hz), 9.23 br.s (1H, NH). Mass spectrum, m/z (I_{rel}, %): 443 (6) $[M + 2]^+$, 442 (10) $[M + 1]^+$, 441 (34) $[M]^+$, 349 (100) $[M - PhNH]^+$, 278 (42), 119 (18), 93 (29) $[PhNH_2]^+$, 77 (14) $[Ph]^+$, 65 (15), 43 (22) $[CHMe_2]^+$. Found, %: C 73.28; H 6.02; N 9.38. $C_{27}H_{27}N_3OS$. Calculated, %: C 73.44; H 6.16; N 9.52. M 484.44.

b. *Compounds **Xa** and **Xb***. Enamine **II**, 1.64 ml (10 mmol), was added to a suspension of 10 mmol of thiopyran **Ia** or **Ig** in 25 ml of anhydrous ethanol, and the mixture was heated for 2 h under reflux. The mixture was allowed to cool down, 5.6 ml (10 mmol) of 10% aqueous potassium hydroxide and 10 mmol of halogen derivative **VIh** or **VIk** was added, the mixture was stirred for 4 h, an additional 5.6 ml (10 mmol) of 10% aqueous potassium hydroxide was added, and the mixture was stirred for 4 h and diluted with an equal volume of water. The precipitate was filtered off and washed with water, ethanol, and hexane. Yield of **Xa** 3.22 g (73%).

3-Amino-4-(2-thienyl)-5,6,7,8-tetrahydrothieno-[2,3-*b*]quinoline-2-carboxamide (Xb). Yield 4.50 g (93%), yellow–green powder, mp 277–278°C (from AcOH; sublimes at 220°C); compound **Xb** shows fluorescence upon UV urradiation. IR spectrum, ν, cm^{-1}: 3202, 3334, 3465 (NH_2); 1668 (CONH); 1645 (δNH_2). ^{1}H NMR spectrum, δ, ppm: 1.70 m (2H, CH_2), 1.84 m (2H, CH_2), 2.52 m (2H, CH_2), 3.00 t (2H, CH_2, J = 6.19 Hz), 6.13 br.s (2H, NH_2), 7.20 d (1H, 3′-H, J = 2.95 Hz), 7.25 d.d (1H, 4′-H, J = 2.40 Hz), 7.35 d and 7.64 d (2H each, C_6H_4, J = 7.08 Hz), 7.79 d (1H, 5′-H, J = 3.71 Hz), 9.24 br.s (1H, NH). Mass spectrum, *m*/*z* (I_{rel}, %): 486 (4) $[M + 2]^+$, 485 (10) $[M + 1]^+$, 484 (11) $[M]^+$, 313 (100) $[M - BrC_6H_4NH]^+$, 171 (9) $[BrC_6H_4NH]^+$, 91 (14), 77 (8) $[Ph]^+$, 40 (35). Found, %: C 54.41; H 3.58; N 8.49. $C_{22}H_{18}BrN_3OS_2$. Calculated, %: C 54.55; H 3.75; N 8.67. *M* 484.43.

3-(4-Bromophenyl)-2,2-pentamethylene-11-(2-thienyl)-1,2,3,4,7,8,9,10-octahydropyrimido-[4′,5′:4,5]thieno[2,3-*b*]quinolin-4-one (XIa). *a*. A mixture of 4.84 g (10 mmol) of compound **Xa** and 1.03 ml (10 mmol) of cyclohexanone in 25 ml of glacial acetic acid was heated for 2 h under reflux. The mixture was cooled, and the yellow crystals were filtered off and washed with glacial acetic acid and diethyl ether. Yield 3.84 g (68%), mp 266–268°C. IR spectrum, ν, cm^{-1}: 3410 (NH), 1712 (C=O). ^{1}H NMR spectrum, δ, ppm: 0.82 m (4H, CH_2), 1.01–1.44 m (6H, CH_2), 1.62–1.97 m (4H, CH_2), 2.11 m (2H, CH_2), 2.99 m (2H, CH_2), 4.58 br.s (1H, NH), 7.14 d and 7.58 d (2H each, C_6H_4, J = 8.00 Hz), 7.32 m (2H, 3′-H, 4′-H), 7.89 d (1H, 5′-H). Found, %: C 59.41; H 4.38; N 7.30. $C_{28}H_{26}BrN_3OS_2$. Calculated, %: C 59.57; H 4.64; N 7.44. *M* 564.57.

b. *Compounds **XIa** and **XIb***. Enamine **II**, 1.64 ml (10 mmol), was added to a suspension of 10 mmol of thiopyran **Ia** or **Ig** in 25 ml of anhydrous ethanol, and the mixture was heated for 2 h under reflux. The mixture was cooled, 5.6 ml (10 mmol) of 10% aqueous potassium hydroxide and 10 mmol of halogen derivative **VIa** or **VIk** were added in succession under stirring, the mixture was stirred for 4 h, an additional 5.6 ml (10 mmol) of 10% aqueous potassium hydroxide was added, and the mixture was stirred for 4 h and diluted with an equal volume of water. The precipitate was filtered off and washed in succession with water, ethanol, and hexane, 1.03 ml (10 mmol) of cyclohexanone and 25 ml of glacial acetic acid were added, and the mixture was heated for 2 h under reflux. After cooling, the precipitate was filtered off and washed with glacial acetic acid and diethyl ether. Yield 4.79 g (95%). The R_f value, IR spectrum, and melting point of **XIa** coincided with the corresponding parameters of a sample synthesized as described above according to method *a*.

11-Isobutyl-2,2-pentamethylene-1,2,3,4,7,8,9,10-octahydropyrimido[4′,5′:4,5]thieno[2,3-*b*]quinolin-4-one (XIb). Yield 3.06 g (80%), white powder, mp 219–221°C (fluoresces under UV irradiation). IR spectrum, ν, cm^{-1}: 3190–3266 (NH), 1684 (CONH). ^{1}H NMR spectrum, δ, ppm: 0.92 d (6H, Me, J = 5.12 Hz), 1.27–1.73 m (8H, CH_2), 1.84 m (2H, CH_2), 2.03 d (2H, C**H**$_2$CH, J = 5.48 Hz), 2.19 m (1H, CH_2C**H**), 2.76 m (2H, CH_2), 2.93 m (6H, CH_2), 5.19 br.s (1H, NH), 7.75 br.s (1H, NHCO). Mass spectrum: *m*/*z* 384 (I_{rel} = 100%) $[M + 1]^+$. Found, %: C 68.69; H 7.48; N 11.12. $C_{22}H_{29}N_3OS$. Calculated, %: C 68.89; H 7.62; N 10.96. *M* 383.56.

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