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Synthesis of 4-Aryl-6-phenyl-2-thioxo-1,2-dihydropyridine-3-carbonitriles by Three-Component Condensation of Aromatic Aldehydes with Cyanothioacetamide and 4,4,4-Trifluoro-1-phenylbutane-1,3-dione

V. D. Dyachenko

Taras Shevchenko Lugansk National University, ul. Oboronnaya 2, Lugansk, 91011 Ukraine
e-mail: dvd_lug@online.lg.ua

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Abstract—4-Aryl-6-phenyl-2-thioxo-1,2-dihydropyridine-3-carbonitriles were synthesized by three-component condensation of aromatic aldehydes with cyanothioacetamide and 4,4,4-trifluoro-1-phenylbutane-1,3-dione. The reaction involved initial formation of Michael adducts which underwent acyl cleavage.

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As shown previously, 3-aryl-2-cyanoprop-2-enethioamides react with 4,4,4-trifluoro-1-phenylbutane-1,3-dione according to the Michael addition pattern, and the subsequent acyl cleavage of the Michael adduct yields 4-aryl-6-phenyl-2-thioxo-1,2-dihydropyridine-3-carbonitriles [1]. 1-(2-Thienyl)-4,4,4-trifluorobutane-1,3-dione behaved similarly in analogous Michael reaction [2].

The present communication reports on three-component condensation of aromatic aldehydes **Ia** and **Ib** with cyanothioacetamide (**II**) and 4,4,4-trifluoro-1-phenylbutane-1,3-dione (**III**). The reactions were carried out in ethanol at 20°C in the presence of *N*-methylmorpholine as catalyst (method *a*), and the products were 4-aryl-6-phenyl-2-thioxo-1,2-dihydropyridine-3-carbonitriles **IVa** and **IVb** (Scheme 1) which attract interest as promising intermediate products for the synthesis of antibacterial [3], analgesic [4], and cardiotonic agents [5], as well as of inhibitors of some enzymes [6].

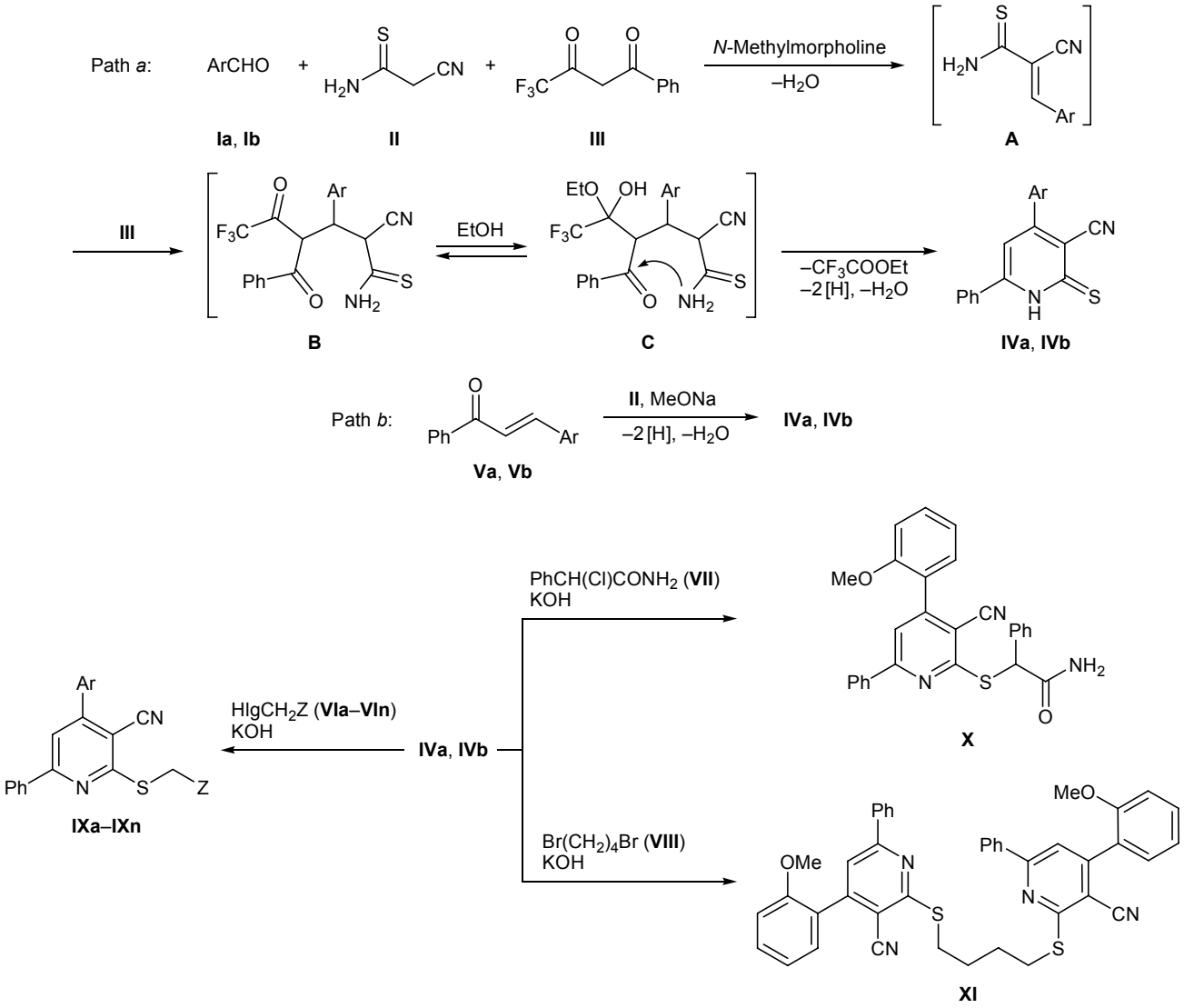
Presumably, the process includes initial formation of Knoevenagel condensation products **A**, followed by Michael addition of cyanothioacetamide (**II**). Adduct **B** thus formed takes up ethanol molecules to produce structure **C** which undergoes acyl cleavage with elimination of ethyl trifluoroacetate [7], and the subsequent intramolecular heterocyclization is accompanied by elimination of water and dehydrogenation. In addition to spectral data [8], the structure of compounds **IVa** and **IVb** was confirmed by their independent synthesis by reaction of chalcones **Va** and **Vb** with cyanothioacetamide (**II**) (method *b*) and by alkylation with alkyl halides **VI**–**VIII** to obtain the corresponding sulfides **IX**–**XI** (Scheme 1).

The IR spectra of compounds **IX**–**XI** contained absorption bands typical of stretching vibrations of conjugated cyano group (2218–2227 cm^{-1}). 2-Thioxo-1,2-dihydropyridine-3-carbonitriles **IX**–**XI** displayed in the ^1H NMR spectra signals from protons in the aromatic substituents, 5-H in the pyridine ring, protons in fragment Z, and methylene protons in the SCH_2 group (δ 2.76–5.84 ppm (see Experimental). In the mass spectra of **IX**–**XI** low-intense peaks from $[M + 2]^+$ ions were observed, which indicated the presence of sulfur in their molecules [9].

EXPERIMENTAL

The IR spectra were recorded on an IKS-40 spectrometer from samples dispersed in mineral oil. The ^1H NMR spectra were obtained on a Bruker AM-300 spectrometer at 300.13 MHz from solutions in DMSO-d_6 using tetramethylsilane as internal reference. The mass spectra (electron impact, 70 eV) were recorded on a Kratos MS-890 instrument with direct sample admission into the ion source. 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

Scheme 1.



I, **IV**, **V**, Ar = 4-$O_2NC_6H_4$ (**a**), 2-$MeOC_6H_4$ (**b**); **VI**, Hlg = Br: Z = 2-MeC_6H_4CO (**a**), 2-oxo-2*H*-chromen-3-ylcarbonyl (**b**), CH_2=$CHCH_2$ (**c**), Et (**i**), 3,4-$Cl_2C_6H_3$ (**l**), 4-BrC_6H_4CO (**m**); Hlg = I: Z = $Me(CH_2)_{10}$ (**d**), H (**j**); Hlg = Cl: Z = PhNHCO (**e**), naphthalen-1-ylcarbamoyl (**f**), HOCO (**g**), H_2NCO (**h**), Ph (**k**), EtOCO (**n**); **IX**, Ar = 4-$O_2NC_6H_4$, Z = 2-MeC_6H_4CO (**a**); Ar = 2-$MeOC_6H_4$: Z = 2-oxo-2*H*-chromen-3-ylcarbonyl (**b**), CH_2=$CHCH_2$ (**c**), $Me(CH_2)_{10}$ (**d**), PhNHCO (**e**), naphthalen-1-ylcarbamoyl (**f**), HOCO (**g**), H_2NCO (**h**), Et (**i**), H (**j**), Ph (**k**), 3,4-$Cl_2C_6H_3$ (**l**), 4-BrC_6H_4CO (**m**), EtOCO (**n**).

acetone–hexane (3:5) as eluent; spots were visualized by treatment with iodine vapor or under UV light.

4-(4-Nitrophenyl)-6-phenyl-2-thioxo-1,2-dihydropyridine-3-carbonitrile (IVa). *a*. Three drops of *N*-methylmorpholine were added at 20°C under stirring to a solution of 10 mmol of 4-nitrobenzaldehyde (**Ia**) and 1.0 g (10 mmol) of cyanothioacetamide (**II**) in 25 ml of ethanol. The mixture was stirred for 15 min, 1.54 ml (10 mmol) of 4,4,4-trifluoro-1-phenylbutane-1,3-dione (**III**) and 2.2 ml (20 mmol) of *N*-methylmorpholine were added, and the mixture was stirred for 2 h, left to stand for 48 h, diluted with 10% hydrochloric acid to pH 5, and left to stand for 24 h. The precipitate was filtered off, washed with ethanol and hexane, and recrystallized from glacial acetic acid. Yield 2.26 g (68%), mp 225–227°C; published data [8]: mp 226–228°C.

4-(2-Methoxyphenyl)-6-phenyl-2-thioxo-1,2-dihydropyridine-3-carbonitrile (IVb) was synthesized in a similar way. Yield 1.97g (62%), yellow powder, mp 138–140°C. IR spectrum, ν, cm^{-1}: 3380 (NH), 2228 (C≡N). ^1H NMR spectrum, δ, ppm: 3.84 s (3H,

Me), 7.08 s (1H, 5-H), 7.12 t (1H, H_{arom}, J = 8.1 Hz), 7.23 d (1H, H_{arom}, J = 8.7 Hz), 7.39–7.63 m (5H, H_{arom}), 7.84 d (1H, H_{arom}, J = 8.1 Hz), 8.03 m (1H, H_{arom}), 14.23 br.s (1H, NH). Found, %: C 71.52; H 4.29; N 8.71. $C_{19}H_{14}N_2OS$. Calculated, %: C 71.68; H 4.43; N 8.80.

Method *b* was described previously [8]; yield of **IVa** 70%, yield of **IVb** 66%.

4-Aryl-2-*Z*-methylsulfanyl-6-phenylpyridine-3-carbonitriles IXa–IXn (*general procedure*). Pyridine-2-thione **IVa** or **IVb**, 10 mmol, was dissolved in 15 ml of DMF, and 5.6 ml (10 mmol) of a 10% aqueous solution of potassium hydroxide and 10 mmol of halogen derivative **VIa**–**VIn** were added in succession. The mixture was stirred for 4 h and diluted with an equal volume of water, and the precipitate was filtered off and washed with water, ethanol, and hexane.

2-(2-Methylbenzoylmethylsulfanyl)-4-(4-nitrophenyl)-6-phenylpyridine-3-carbonitrile (IXa). Yield 3.44 g (74%), yellow powder, mp 171–173°C (from AcOH). IR spectrum, ν, cm^{-1}: 2225 (C≡N), 1711 (C=O). NMR spectrum ^{1}N, δ, ppm: 2.98 s (3H, Me), 4.96 s (2H, CH_2), 7.19 t (2H, H_{arom}, J = 7.14 Hz), 7.34 t (1H, H_{arom}, J = 6.72 Hz), 7.55 t (2H, H_{arom}, J = 7.24 Hz), 7.65 t (1H, H_{arom}, J = 6.72 Hz), 7.81 s (1H, 5-H), 7.85 d (2H, H_{arom}, J = 8.08 Hz), 7.98 d (2H, H_{arom}, J = 8.68 Hz), 8.11 d (2H, H_{arom}, J = 7.36 Hz), 8.39 d (2H, H_{arom}, J = 8.68 Hz). Mass spectrum, *m*/*z* (I_{rel}, %): 451 (49) [M – Me]$^+$, 434 (18), 346 (62) [M – Me – C_6H_5CO]$^+$, 330 (40), 300 (15), 227 (19), 105 (100) [C_6H_5CO]$^+$, 91 (4) [$PhCH_2$]$^+$, 77 (38) [Ph]$^+$, 51 (5). Found, %: C 69.50; H 4.02; N 8.95. $C_{27}H_{19}N_3O_3S$. Calculated, %: C 69.66; H 4.11; N 9.03. M 465.5.

4-(2-Methoxyphenyl)-2-(2-oxo-2*H*-chromen-3-ylcarbonylmethylsulfanyl)-6-phenylpyridine-3-carbonitrile (IXb). Yield 3.93 g (78%), colorless crystals, mp 192–194°C (from BuOH). IR spectrum, ν, cm^{-1}: 2228 (C≡N); 1714, 1695 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.82 s (3H, Me), 5.01 s (2H, CH_2), 7.02–7.61 m (10H, H_{arom}), 7.72–8.12 m (4H, H_{arom}), 8.69 s (1H, 4′-H). Found, %: C 71.35; H 3.88; N 5.39. $C_{30}H_{20}N_2O_4S$. Calculated, %: C 71.41; H 4.00; N 5.55.

2-Allylsulfanyl-4-(2-methoxyphenyl)-6-phenylpyridine-3-carbonitrile (IXc). Yield 2.83 g (79%), mp 88–89°C (from BuOH). IR spectrum: ν 2222 cm^{-1} (C≡N). ^{1}H NMR spectrum, δ, ppm: 3.81 s (3H, Me), 4.11 d (2H, SCH_2, J = 6.57 Hz), 5.21 d (1H, =CH_2, J_{cis} = 9.55 Hz), 5.47 d (1H, =CH_2, J_{trans} = 17.3 Hz), 5.81–6.22 m (1H, CH=), 7.02–7.69 m (7H, H_{arom}), 7.84 s (1H, 5-H), 8.25 m (2H, H_{arom}). Found, %: C 73.68; H 4.95; N 7.66. $C_{22}H_{18}N_2OS$. Calculated, %: C 73.72; H 5.06; N 7.81.

2-Dodecylsulfanyl-4-(2-methoxyphenyl)-6-phenylpyridine-3-carbonitrile (IXd). Yield 3.21 g (66%), mp 55–56°C (from MeOH). IR spectrum: ν 2226 cm^{-1} (C≡N). ^{1}H NMR spectrum, δ, ppm: 0.87 t (3H, Me, J = 6.5 Hz), 1.18–1.62 m (18H, CH_2), 1.81 m (2H, CH_2), 3.36 t (2H, SCH_2, J = 7.24 Hz), 3.85 s (3H, MeO), 7.12 t (2H, H_{arom}, J = 7.32 Hz), 7.31 d (1H, H_{arom}, J = 7.32 Hz), 7.43–7.53 m (4H, H_{arom}), 7.61 s (4H, 5-H), 8.15 m (2H, H_{arom}). Mass spectrum, *m*/*z* (I_{rel}, %): 488 (2) [M + 2]$^+$, 487 (19) [M + 1]$^+$, 486 (63) [M]$^+$, 485 (51) [M – H]$^+$, 471 (92) [M – Me]$^+$, 455 (54), 318 (87), 301 (62), 287 (22), 214 (10), 57 (36), 43 (100). Found, %: C 76.38; H 7.75; N 5.60. $C_{31}H_{38}N_2OS$. Calculated, %: C 76.50; H 7.87; N 5.76. M 486.7.

[3-Cyano-4-(2-methoxyphenyl)-6-phenylpyridin-2-ylsulfanyl]-*N*-phenylacetamide (IXe). Yield 3.2 g (71%), mp 185–187°C (from BuOH). IR spectrum, ν, cm^{-1}: 3332 (NH), 2220 (C≡N), 1671 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.83 s (3H, Me), 4.23 s (2H, CH_2), 6.97–7.63 m (13H, H_{arom}), 8.08 d (2H, H_{arom}, J = 7.82 Hz), 10.24 br.s (1H, NH). Mass spectrum, *m*/*z* (I_{rel}, %): 453 (2) [M + 2]$^+$, 452 (8) [M + 1]$^+$, 451 (25) [M]$^+$, 436 (10) [M – Me]$^+$, 359 (92) [M – PhNH]$^+$, 331 (100) [PhNHCO]$^+$, 317 (86), 301 (89), 132 (22), 93 (28) [$PhNH_2$]$^+$, 77 (29) [Ph]$^+$, 65 (26), 39 (7). Found, %: C 71.72; H 4.50; N 9.22. $C_{27}H_{21}N_3O_2S$. Calculated, %: C 71.82; H 4.69; N 9.31. M 451.6.

[3-Cyano-4-(2-methoxyphenyl)-6-phenylpyridin-2-ylsulfanyl]-*N*-(naphthalen-1-yl)acetamide (IXf). Yield 3.46 g (69%), mp 225–227°C (from DMF). IR spectrum, ν, cm^{-1}: 3341 (NH), 2227 (C≡N), 1666 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.87 s (3H, Me), 4.42 s (2H, CH_2), 7.07–7.18 m (2H, H_{arom}), 7.30–7.54 m (8H, H_{arom}), 7.65 d (1H, H_{arom}, J = 7.48 Hz), 7.68 s (1H, 5-H), 7.75 d (1H, H_{arom}, J = 7.58 Hz), 7.84 d (1H, H_{arom}, J = 8.02 Hz), 8.08 d (1H, H_{arom}, J = 8.0 Hz), 8.21 d (1H, H_{arom}, J = 7.38 Hz), 10.19 br.s (1H, NH). Mass spectrum, *m*/*z* (I_{rel}, %): 501 (3) [M + 1]$^+$, 501 (19) [M]$^+$, 359 (100) [M – $C_{10}H_8N$]$^+$, 331 (85), 31 (68), 301 (57), 287 (12), 183 (32), 143 (95) [1-aminonaphthalene]$^+$, 115 (91), 43 (6). Found, %: C 74.11; H 4.58; N 8.29. $C_{31}H_{23}N_3O_2S$. Calculated, %: C 74.23; H 4.62; N 8.38. M 501.6.

[3-Cyano-4-(2-methoxyphenyl)-6-phenylpyridin-2-ylsulfanyl]acetic acid (IXg). Yield 2.86 g (76%), mp 160–162°C (from AcOH). IR spectrum, ν, cm^{-1}:

2217 (C≡N), 1720 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.83 s (3H, Me), 4.18 s (2H, CH_2), 7.02–7.35 m (3H, H_{arom}), 7.49–7.61 m (4H, H_{arom}), 7.80 s (1H, 5-H), 8.23 m (2H, H_{arom}), 12.96 br.s (1H, OH). Found, %: C 66.89; H 4.05; N 7.30. $C_{21}H_{16}N_2O_3S$. Calculated, %: C 67.01; H 4.28; N 7.44.

[3-Cyano-4-(2-methoxyphenyl)-6-phenylpyridin-2-ylsulfanyl]acetamide (IXh). Yield 2.63 g (70%), mp 203–209°C (from BuOH). IR spectrum, ν, cm^{-1}: 3298 (NH_2), 2225 (C≡N), 1672 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.82 s (3H, Me), 4.10 s (2H, CH_2), 7.05–7.61 m (8H, H_{arom}, NH_2), 7.76 br.s (1H, NH_2), 7.84 s (1H, 5-H), 8.29 m (2H, H_{arom}). Found, %: C 67.03; H 4.41; N 11.05. $C_{21}H_{17}N_3O_2S$. Calculated, %: C 67.18; H 4.56; N 11.19.

4-(2-Methoxyphenyl)-6-phenyl-2-propylsulfanylpyridine-3-carbonitrile (IXi). Yield 2.45 g (68%), mp 101–103°C (from MeOH). IR spectrum: ν 2219 cm^{-1} (C≡N). ^{1}H NMR spectrum, δ, ppm: 1.05 t (3H, Me, J = 7.11 Hz), 1.78 m (2H, CH_2), 3.28 t (2H, SCH_2, J = 7.19 Hz), 3.81 s (3H, MeO), 7.05–7.64 m (7H, H_{arom}), 7.81 s (1H, 5-H), 8.22 m (2H, H_{arom}). Found, %: C 73.18; H 5.42; N 7.60. $C_{22}H_{20}N_2OS$. Calculated, %: C 73.30; H 5.59; N 7.77.

4-(2-Methoxyphenyl)-2-methylsulfanyl-6-phenylpyridine-3-carbonitrile (IXj). Yield 2.49 g (75%), mp 143–145°C (from BuOH). IR spectrum: ν 2220 cm^{-1} (C≡N). ^{1}H NMR spectrum, δ, ppm: 2.76 s (3H, MeS), 3.81 s (3H, MeO), 7.02–7.68 m (7H, H_{arom}), 7.83 s (1H, 5-H), 8.27 m (2H, H_{arom}). Found, %: C 72.12; H 4.71; N 8.34. $C_{20}H_{16}N_2OS$. Calculated, %: C 72.26; H 4.85; N 8.43.

2-Benzylsulfanyl-4-(2-methoxyphenyl)-6-phenylpyridine-3-carbonitrile (IXk). Yield 3.0 g (74%), mp 135–137°C (from EtOH). IR spectrum: ν 2226 cm^{-1} (C≡N). ^{1}H NMR spectrum, δ, ppm: 3.86 s (3H, Me), 4.60 s (2H, CH_2), 7.05–7.16 m (2H, H_{arom}), 7.23–7.34 m (4H, H_{arom}), 7.41–7.5 m (6H, H_{arom}), 7.63 s (1H, 5-H), 8.12 m (2H, H_{arom}). Found, %: C 76.31; H 4.80; N 6.75. $C_{26}H_{20}N_2OS$. Calculated, %: C 76.44; H 4.94; N 6.86.

2-(3,4-Dichlorobenzoylmethylsulfanyl)-4-(2-methoxyphenyl)-6-phenylpyridine-3-carbonitrile (IXl). Yield 3.48 g (69%), mp 151–153°C (from AcOH). IR spectrum, ν, cm^{-1}: 2222 (C≡N), 1712 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.82 s (3H, Me), 5.05 s (2H, CH_2), 7.01–7.62 m (6H, H_{arom}), 7.74–7.86 m (4H, H_{arom}), 8.02 d (1H, H_{arom}, J = 6.95 Hz), 8.12 d (1H, H_{arom}, J = 6.95 Hz), 8.34 d (1H, H_{arom}, J = 7.14 Hz). Found, %: C 64.03; H 3.44; N 5.39. $C_{27}H_{18}Cl_2N_2O_2S$. Calculated, %: C 64.16; H 3.59; N 5.54.

2-(4-Bromobenzoylmethylsulfanyl)-4-(2-methoxyphenyl)-6-phenylpyridine-3-carbonitrile (IXm). Yield 4.17 g (81%), mp 159–161°C (from BuOH). IR spectrum, ν, cm^{-1}: 2224 (C≡N), 1714 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.83 s (3H, Me), 5.03 s (2H, CH_2), 7.04–7.69 m (8H, H_{arom}), 7.81 d (4H, H_{arom}, J = 7.8 Hz), 8.08 d (2H, H_{arom}, J = 7.04 Hz). Found, %: C 62.83; H 3.61; N 5.33. $C_{27}H_{19}BrN_2O_2S$. Calculated, %: C 62.92; H 3.72; N 5.44.

Ethyl [3-cyano-4-(2-methoxyphenyl)-6-phenylpyridin-2-ylsulfanyl]acetate (IXn). Yield 3.15 g (78%), mp 170–172°C (from EtOH). IR spectrum, ν, cm^{-1}: 2219 (C≡N), 1715 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.18 t (3H, Me, J = 6.18 Hz), 3.82 s (3H, MeO), 4.11 q (2H, OCH_2, J = 6.18 Hz), 4.26 s (2H, SCH_2), 7.03–7.63 m (7H, H_{arom}), 7.87 s (1H, 5-H), 8.20 m (2H, H_{arom}). Found, %: C 68.19; H 4.78; N 6.88. $C_{23}H_{20}N_2O_3S$. Calculated, %: C 68.30; H 4.98; N 6.93.

[3-Cyano-4-(2-methoxyphenyl)-6-phenylpyridin-2-ylsulfanyl]phenylacetamide (X) was synthesized as described for compounds **IX** from 3.18 g (10 mmol) of pyridinethione **IVb** and 1.7 g (10 mmol) of chloro(phenyl)acetamide (**VII**). Yield 3.07 g (68%), mp 240–244°C (from BuOH) (sublimes at 210°C). IR spectrum, ν, cm^{-1}: 2225 (C≡N), 1668 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.81 s (3H, Me), 5.84 s (1H, SCH), 7.02–7.73 m (13H, H_{arom}, NH_2), 7.83 s (1H, 5-H), 8.05 br.s (1H, NH_2), 8.28 m (2H, H_{arom}). Found, %: C 71.69; H 4.58; N 9.22. $C_{27}H_{21}N_3O_2S$. Calculated, %: C 71.82; H 4.69; N 9.31.

2,2′-(Butane-1,4-diylbissulfanyl)bis[(2-methoxyphenyl)-6-phenylpyridine-3-carbonitrile] (XI) was synthesized as described for compounds **IX** from 3.18 g (10 mmol) of pyridinethione **IVb** and 0.59 ml (5 mmol) of 1,4-dibromobutane (**VIII**). Yield 1.51 g (48%), mp 188–189°C (from MeCN). IR spectrum: ν 2228 cm^{-1} (C≡N). ^{1}H NMR spectrum, δ, ppm: 2.01 m (4H, CH_2), 3.46 m (4H, SCH_2), 3.81 s (6H, OMe), 6.95–7.68 m (14H, H_{arom}), 7.79 s (2H, 5-H), 8.18 m (4H, H_{arom}). Found, %: C 80.14; H 4.03; N 8.82. $C_{42}H_{26}N_4O_2S_2$. Calculated, %: C 80.22; H 4.17; N 8.91.

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