ISSN 1070-4280, Russian Journal of Organic Chemistry, 2014, Vol. 50, No. 12, pp. 1821–1825. © Pleiades Publishing, Ltd., 2014.
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Synthesis of 4-Methylmorpholinium 6-Amino-3,5-dicyano-4-(furan-2-yl)pyridine-2-thio(seleno)lates and 3-[Aryl(hetaryl)]-2-cyanoprop-2-enethioamides by Michael Reaction

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Received July 16, 2014

Abstract—Michael reaction of dimethyl (furan-2-ylmethylidene)malonate with 2-cyanoethanethio(seleno)-amides and 4-methylmorpholine afforded 4-methylmorpholinium 6-amino-3,5-dicyano-4-(furan-2-yl)pyridine-2-thio(seleno)lates via exchange of the CH acid components. Aryl(hetaryl)methylidenemalononitriles reacted with cyanoethanethioamide under analogous conditions to give 3-aryl(hetaryl)-2-cyanoprop-2-enethioamides which were converted into 3-aryl(hetaryl)-2-(1,3-thiazol-2-yl)prop-2-enenitriles according to Hantzsch.

DOI: 10.1134/S1070428014120185

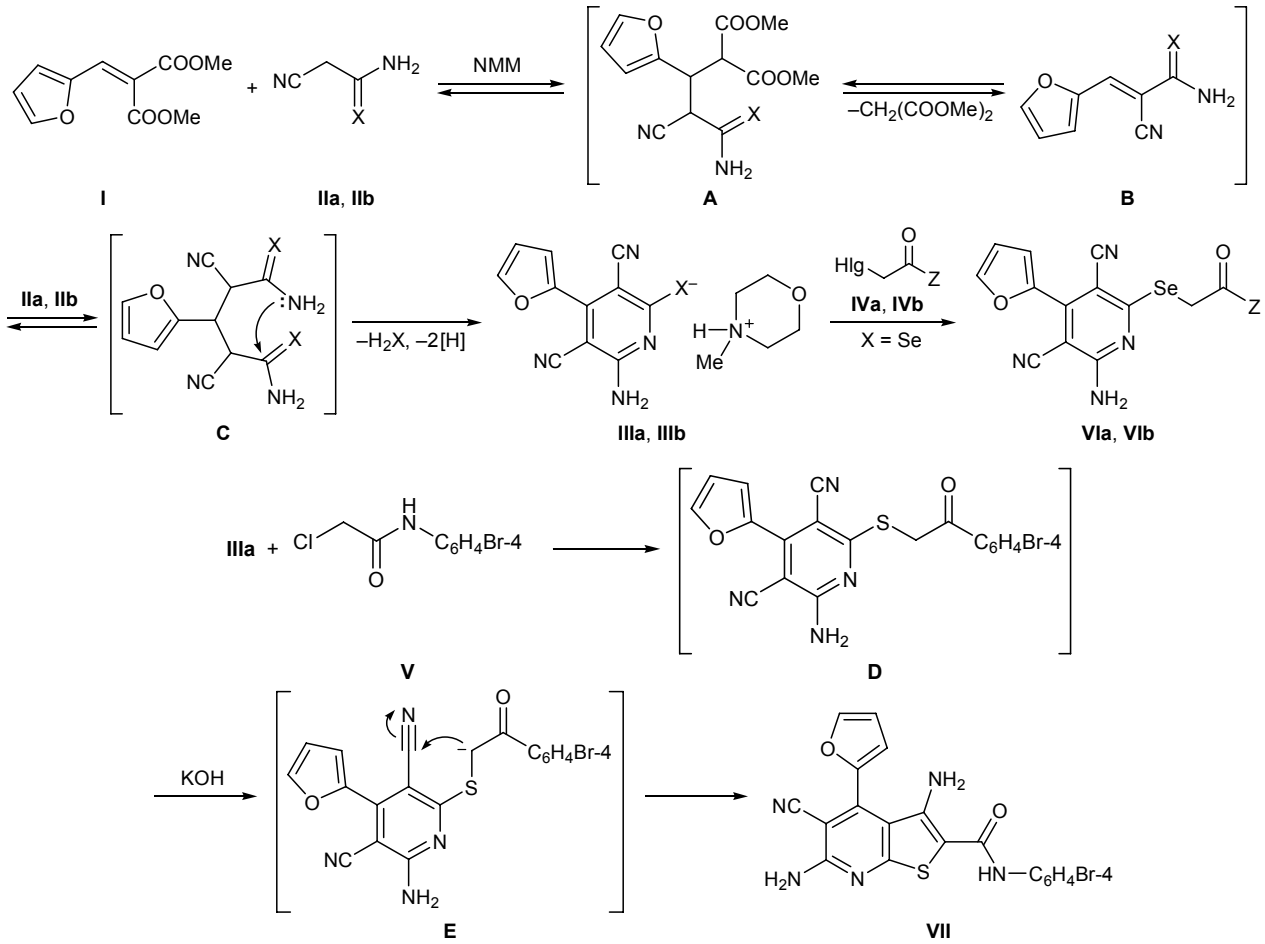
Michael reaction is widely used in the synthesis of carbo- and heterocycles [1]. One of its versions involving exchange of CH acid components has been reported in a few publications [2]. This path is typical of reactions of cyanoacetohydrazides with cinnamonitriles [3], cyanoacetanilides with 2-cyano-3-(furan-2-yl)-prop-2-enethioamide [4], 2-(benzothiazol-2-yl)-ethanenitrile with (pyrrol-2-ylmethylidene)malononitrile [5], malononitrile with 2-cyano-3-cycloalkylprop-2-enamides [6], 2-cyanoethanamide with 2-benzoyl-3-(4-nitrophenyl)prop-2-enenitrile [7], 2-[1-aryl-(hetaryl)ethylidene]malononitriles with 3-aryl-(hetaryl)-2-cyanoprop-2-enethioamides [8], and 2-cyanoethanethioamide with diethyl benzylidenemalonate or 3-benzylidenepyrazol-2-one [9].

The present article describes new Michael reactions following the above pattern. Dimethyl (furan-2-ylmethylidene)malonate (**I**) reacted with 2-cyanoethanethioamide (**IIa**) and 2-cyanoethaneselenoamide (**IIb**) in anhydrous ethanol in the presence of an equimolar amount of *N*-methylmorpholine at 20°C under argon to give 4-methylmorpholinium 6-amino-3,5-dicyano-4-(furan-2-yl)pyridine-2-thio(seleno)lates **IIIa** and **IIIb** (Scheme 1). Presumably, initially formed Michael adduct **A** loses dimethyl malonate molecule with formation of intermediate **B**. The latter then acts as Michael acceptor toward initial CH acid **II** (Michael donor) to produce new Michael adduct **C** whose chemoselective intramolecular cyclization yields substituted pyridine **III**. Compounds **IIIa** and **IIIb** were isolated as stable 4-methylmorpholinium salts which were identified by comparing their spectral parameters with published data [10, 11]. In addition, the alkylation of **IIIa** and **IIIb** with halomethyl compounds **IVa** and **IVb** and *N*-(4-bromophenyl)-2-chloroethanamide (**V**) led to the formation of selenides **VIa** and **VIb** and thieno[2,3-*b*]pyridine derivative **VII**. Compound **VII** was formed as a result of intramolecular cyclization of intermediate **D** through carbanion **E**. The proposed scheme for the formation of morpholinium salts **IIIa** and **IIIb** is confirmed by the fact that their yield increased when 2 equiv of CH acid **IIa** or **IIb** was used.

The structure of compounds **VIa**, **VIb**, and **VII** was confirmed by spectral data. In the IR spectra of **VIa**, **VIb**, and **VII**, absorption bands due to stretching vibrations of the carbonyl and conjugated cyano groups were located at 1711–1714 and 2218–2224 cm^{-1}, respectively. The ^1H NMR spectra of **VIa** and **VIb** contained signals from protons in the amino group, furan ring, and $SeCH_2C(O)Z$ fragment (see Experimental). Compound **VII** showed in the ^1H NMR spectrum signals from protons in the two amino groups as broadened singlets at δ 6.34 (3-NH_2) and 7.55 ppm (6-NH_2), which is typical of such systems [12].

Under analogous conditions, the Michael reaction of 2-cyanoethanethioamide (**IIa**) with [aryl(hetaryl)-

Scheme 1.



NMM is *N*-methylmorpholine; **II**, **III**, X = S (**a**), Se (**b**); **IV**, Hlg = Cl, Z = OCH_2Ph (**a**); Hlg = Br, Z = 2-oxo-2*H*-chromen-3-yl (**b**); **VI**, Z = OCH_2Ph (**a**), 2-oxo-2*H*-chromen-3-yl (**b**).

methylidene]malononitriles **VIIIa**–**VIIIe** also involved exchange of the methylene components (path *a* in Scheme 2) and produced 3-aryl(hetaryl)-2-cyanoprop-2-enethioamides **IXa**–**IXe**. Presumably, elimination of malononitrile from primary Michael adduct **F** yields poorly soluble unsaturated thioamide **IX**, and the equilibrium is displaced toward the latter.

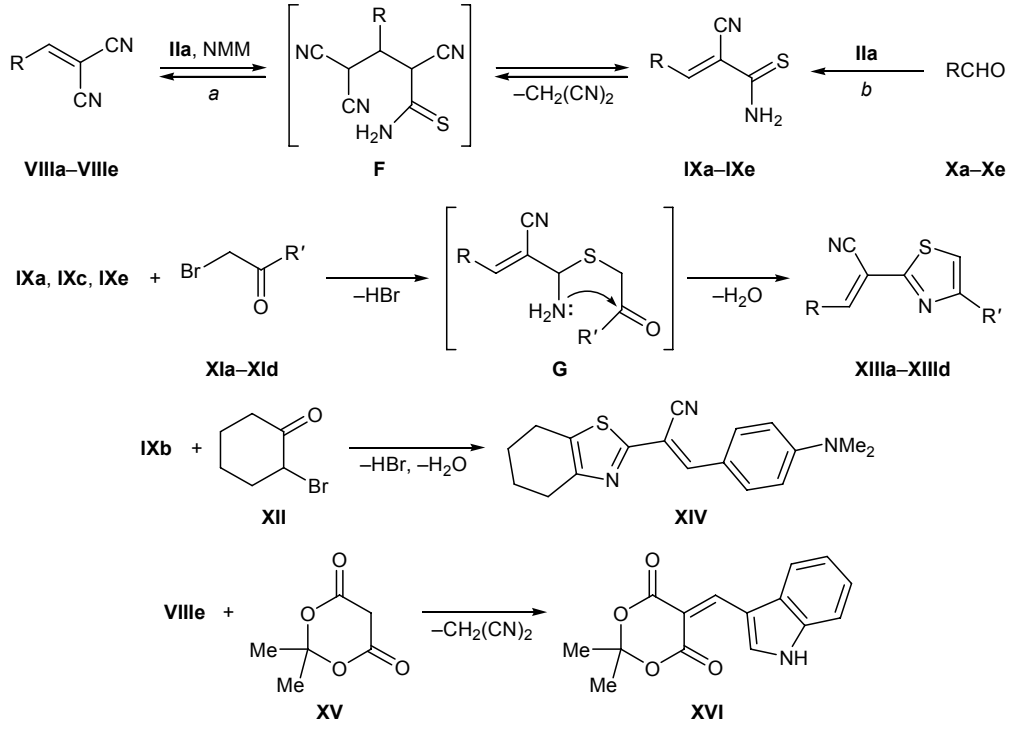
The structure of **IXa**–**IXe** was consistent with their spectral parameters (see Experimental) and was also confirmed by chemical transformations and independent synthesis from aromatic aldehydes **Xa**–**Xe** and cyanothioacetamide (**IIa**) according to Knoevenagel (path *b*). As a qualitative test for thioamide group, compounds **IXa**–**IXe** were brought into the Hantzsch condensation with α-halocarbonyl compounds **XIa**–**XId** and **XII**, which afforded the corresponding substituted thiazoles **XIIIa**–**XIIId** and **XIV** through intermediate sulfides **G**. Thiazoles **XIIIa**–**XIIId** and **XIV** attract interest as potential pharmaceutical agents [13].

Likewise, the reaction of (1*H*-indol-3-ylmethylidene)malononitrile (**VIIIe**) with 2,2-dimethyl-1,3-dioxan-4,6-dione (**XV**, Meldrum's acid) in ethanol at 20°C in the presence of an equimolar amount of 4-methylmorpholine gave previously unknown Meldrum's acid derivative **XVI**. Compounds **XIIIa**–**XIIId**, **XIV**, **XVI** characteristically showed in the ^{1}H NMR spectra a singlet from the vinylic proton at δ 7.66–8.55 ppm.

EXPERIMENTAL

The IR spectra were recorded on an IKS-40 spectrometer from samples dispersed in mineral oil. The ^{1}H NMR spectra were measured on a Varian Mercury-400 instrument at 400.397 MHz using DMSO-d_6 as solvent and tetramethylsilane as internal reference. The mass spectra (electron impact, 70 eV) were obtained on a Hewlett Packard 5890/5972 GC/MS system (HP-5 MS column); samples were injected as solutions

Scheme 2.



NMM is *N*-methylmorpholine; **VIII**–**X**, R = 3,4-$(MeO)_2C_6H_3$ (**a**), 4-$Me_2HC_6H_4$ (**b**), 5-phenylfuran-2-yl (**c**), 5-bromofuran-2-yl (**d**), 1*H*-indol-3-yl (**e**); **XI**, R′ = Ph (**a**), thiophen-2-yl (**b**), 4-MeC_6H_4 (**c**), Me_2CHCH_2 (**d**); **XIII**, R = 3,4-$(MeO)_2C_6H_3$, R′ = Me_2CHCH_2 (**a**); R = 5-phenylfuran-2-yl, R′ = 4-MeC_6H_4 (**b**), thiophen-2-yl (**c**); R = 1*H*-indol-3-yl, R′ = Ph (**d**).

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 UV254 plates using acetone–hexane (3:5) as eluent; spots were detected by treatment with iodine vapor or under UV light.

4-Methylmorpholinium 6-amino-3,5-dicyano-4-(furan-2-yl)pyridine-2-thiolate (IIIa). Dimethyl (furan-2-ylmethylidene)malonate (**I**), 2.1 g (10 mmol), was dissolved in 25 mL of anhydrous ethanol, 2.0 g (20 mmol) of CH acid **IIa** and 1.1 mL (10 mmol) of 4-methylmorpholine were added under stirring at 20°C, and the mixture was stirred for 15 min until it became homogeneous and left to stand for 48 h. The precipitate was filtered off and washed with ethanol and hexane. Yield 2.2 g (63%), mp 233–234°C; published data [11]: mp 230–232°C.

4-Methylmorpholinium 6-amino-3,5-dicyano-4-(furan-2-yl)pyridine-2-selenolate (IIIb) was synthesized in a similar way from 2.9 g (20 mmol) of 2-cyanoethaneselenoamide (**IIb**). Yield 2.3 g (60%), mp 317–320°C; published data [10]: mp 320–323°C.

Benzyl 2-[6-amino-3,5-dicyano-4-(furan-2-yl)-pyridin-2-ylselanyl]ethanoate (VIa). A mixture of 3.9 g (10 mmol) of morpholinium salt **IIIb** and 1.52 mL (10 mmol) of ester **IVa** in 15 mL of DMF was stirred for 5 h at 20°C. The mixture was diluted with an equal volume of water, and the precipitate was filtered off and washed with water, ethanol, and hexane. Yield 3.1 g (72%), violet needles, mp 192–194°C (from EtOH). IR spectrum, ν, cm^{-1}: 3384, 3311, 2910 (NH_2); 2222 (C≡N), 1714 (C=O), 1645 (δNH_2). ^{1}H NMR spectrum, δ, ppm: 4.24 s (2H, OCH_2), 5.16 s (2H, $SeCH_2$), 7.65 s (1H, 3′-H), 7.30–7.39 m (5H, Ph), 7.44 s (1H, 4′-H), 8.00 br.s (2H, NH_2), 8.11 s (1H, 5′-H). Mass spectrum: *m*/*z* 438 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 54.80; H 3.14; N 12.72. $C_{20}H_{14}N_4O_3Se$. Calculated, %: C 54.93; H 3.23; N 12.81.

2-Amino-4-(furan-2-yl)-6-[2-oxo-2-(2-oxo-2*H*-chromen-3-yl)ethylselanyl]pyridine-3,5-dicarbonitrile (VIb) was synthesized as described above for compound **VIa** from 2.7 g (10 mmol) of coumarin derivative **IVb**. Yield 4.4 g (92%), colorless powder, mp 245–247°C (from BuOH). IR spectrum, ν, cm^{-1}: 3388, 3300, 3210 (NH_2), 2218 (C≡N), 1711 (C=O), 1648 (δNH_2). ^{1}H NMR spectrum, δ, ppm: 4.64 s (2H, $SeCH_2$), 6.58 s (1H, 3′-H), 7.18–7.32 m (5H, H_{arom}), 7.34 t (1H, H_{arom}, *J* = 7.5 Hz), 7.95 br.s (2H, NH_2),

8.63 s (1H, 4″-H). Mass spectrum: *m*/*z* 476 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 55.40; H 2.41; N 11.65. $C_{22}H_{12}N_4O_4Se$. Calculated, %: C 55.59; H 2.55; N 11.79.

3,6-Diamino-*N*-(4-bromophenyl)-5-cyano-4-(furan-2-yl)thieno[2,3-*b*]pyridine-2-carboxamide (VII). To a solution of 3.43 g (10 mmol) of morpholinium salt **IIIa** and 2.5 g (10 mmol) of *N*-(4-bromophenyl)-2-chloroethanamide (**V**) in 20 mL of DMF we added under stirring at 20°C 5.6 mL (10 mmol) of 10% aqueous potassium hydroxide. 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. Yield 3.7 g (81%), yellow powder, mp 263–265°C (from BuOH). IR spectrum, ν, cm^{-1}: 3395, 3311, 3198 (NH_2); 2224 (C≡N), 1672 (C=O), 1649 (δNH_2). ^{1}H NMR spectrum, δ, ppm: 6.34 br.s (2H, 3-NH_2), 6.89 d (1H, 3′-H, *J* = 2.9 Hz), 7.13 d.d (1H, 4′-H, *J* = 2.4, 1.1 Hz), 7.47 d (2H, H_{arom}, *J* = 7.5 Hz), 7.55 br.s (2H, 6-NH_2), 7.68 d (2H, H_{arom}, *J* = 7.5 Hz), 8.09 d (1H, 5′-H, *J* = 1.1 Hz), 10.49 br.s (1H, NHCO). Mass spectrum: *m*/*z* 455 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 50.11; H 2.58; N 15.32. $C_{19}H_{12}BrN_5O_2S$. Calculated, %: C 50.23; H 2.66; N 15.42.

Compounds IXa–IXe (*general procedures*). *a*. A mixture of 10 mmol of [aryl(hetaryl)methylidene]-malononitrile **VIIIa–VIIIe**, 1.0 g (10 mmol) of 2-cyanoethanethioamide (**IIa**), and 1.1 mL (10 mmol) of 4-methylmorpholine in 20 mL of ethanol was stirred for 5 h at 20°C and was left to stand for 24 h. The precipitate was filtered off and washed with ethanol and hexane.

b. A mixture of 10 mmol of aromatic aldehyde **Xa–Xe**, 1.0 g (10 mmol) of 2-cyanoethanethioamide (**IIa**), and three drops of 4-methylmorpholine in 20 mL of ethanol was stirred for 5 h at 20°C and was then left to stand for 24 h. The precipitate was filtered off and washed with ethanol and hexane.

2-Cyano-3-(3,4-dimethoxyphenyl)prop-2-enethioamide (IXa). Yield 2.0 g (82%) (*a*), 75% (*b*); yellow crystals, mp 198–200°C (from PhH); published data [14]: mp 197–201°C.

2-Cyano-3-(4-dimethylaminophenyl)prop-2-enethioamide (IXb). Yield 1.8 g (78%) (*a*), 84% (*b*); yellow crystals, mp 230–232°C (from EtOH); published data [15]: mp 231–232°C. IR spectrum, ν, cm^{-1}: 3330, 3218, 3177 (NH_2); 2200 (C≡N). ^{1}H NMR spectrum, δ, ppm: 3.07 s (6H, Me), 6.83 d and 7.87 d (2H each, H_{arom}, *J* = 8.2 Hz), 8.07 s (1H, CH=), 9.12 br.s and 9.66 br.s (1H each, NH_2). Mass spectrum: *m*/*z* 232 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 62.22; H 5.55; N 18.08. $C_{12}H_{13}N_3S$. Calculated, %: C 62.31; H 5.66; N 18.17.

2-Cyano-3-(5-phenylfuran-2-yl)prop-2-enethioamide (IXc). Yield 1.8 g (70%) (*a*), 77% (*b*); red crystals, mp 153–155°C (from EtOH). IR spectrum, ν, cm^{-1}: 2188–3395 (NH_2), 2225 (C≡N). ^{1}H NMR spectrum, δ, ppm: 7.14–7.49 m (4H, H_{arom}), 7.61–8.02 m (3H, H_{arom}), 8.19 s (1H, CH=), 9.41 br.s and 10.02 br.s (1H each, NH_2). Mass spectrum: *m*/*z* 255 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 66.02; H 3.84; N 10.95. $C_{14}H_{10}N_2OS$. Calculated, %: C 66.12; H 3.96; N 11.02.

3-(5-Bromofuran-2-yl)-2-cyanoprop-2-enethioamide (IXd). Yield 2.0 g (77%) (*a*), 86% (*b*); yellow powder, mp 148–150°C (from EtOH). IR spectrum, ν, cm^{-1}: 3195–3312 (NH_2), 2202 (C≡N). ^{1}H NMR spectrum, δ, ppm: 6.46 d (1H, 3′-H, *J* = 3.1 Hz), 7.43 d (1H, 4′-H, *J* = 3.1 Hz), 7.94 s (1H, CH=), 9.38 br.s and 10.01 br.s (1H each, NH_2). Mass spectrum: *m*/*z* 258 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 37.28; H 1.85; N 10.85. $C_8H_5BrN_2OS$. Calculated, %: C 37.37; H 1.96; N 10.90.

2-Cyano-3-(1*H*-indol-3-yl)prop-2-enethioamide (IXe). Yield 1.7 g (75%) (*a*), 85% (*b*); yellow powder, mp 173–175°C (from EtOH); published data [16]: mp 174–176°C.

Compounds XIIIa–XIIId (*general procedure*). A mixture of 10 mmol of thioamide **IXa–IXd** and 10 mmol of α-bromo ketone **XIa–XId** in 15 mL of DMF was stirred for 3 h at 20°C, diluted with an equal volume of water, and left to stand for 24 h. The precipitate was filtered off and washed with water, ethanol, and hexane.

3-(3,4-Dimethoxyphenyl)-2-(4-isobutyl-1,3-thiazol-2-yl)prop-2-enenitrile (XIIIa). Yield 2.3 g (69%), yellow powder, mp 68–70°C (from EtOH). IR spectrum: ν 2214 cm^{-1} (C≡N). ^{1}H NMR spectrum, δ, ppm: 0.95 d (6H, Me, *J* = 6.5 Hz), 2.01–2.18 m (1H, C**H**Me_2), 2.60 d (2H, CH_2, *J* = 7.0 Hz), 3.87 s and 3.88 s (3H each, MeO), 8.97 d (1H, H_{arom}, *J* = 8.0 Hz), 7.05 s (1H, H_{arom}), 7.45 d (1H, H_{arom}, *J* = 8.0 Hz), 7.66 s (1H, CH=), 8.02 s (1H, 5″-H). Mass spectrum: *m*/*z* 329 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 65.70; H 6.02; N 8.47. $C_{18}H_{20}N_2O_2S$. Calculated, %: C 65.83; H 6.14; N 8.53.

2-[4-(4-Methylphenyl)-1,3-thiazol-2-yl]-3-(5-phenylfuran-2-yl)prop-2-enenitrile (XIIIb). Yield 2.9 g (79%), yellow crystals, mp 167–169°C (from BuOH).

IR spectrum: ν 2213 cm^{-1} (C≡N). ^1H NMR spectrum, δ, ppm: 2.38 s (3H, Me), 7.21–7.58 m (6H, H_{arom}), 7.81–8.02 m (5H, H_{arom}), 8.06 s (1H, CH=), 8.11 s (1H, 5″-H). Mass spectrum: *m*/*z* 369 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 74.89; H 4.25; N 7.52. $C_{23}H_{16}N_2OS$. Calculated, %: C 74.98; H 4.38; N 7.60.

3-(5-Phenylfuran-2-yl)-2-[4-(thiophen-2-yl)-1,3-thiazol-2-yl]prop-2-enenitrile (XIIIc). Yield 2.9 g (80%), yellow powder, mp 143–145°C (from BuOH). IR spectrum: ν 2210 cm^{-1} (C≡N). ^1H NMR spectrum, δ, ppm: 7.02–7.18 m (2H, H_{arom}), 7.32–7.68 m (7H, H_{arom}), 7.85–7.93 m (2H, H_{arom}), 8.05 s (1H, CH=). Mass spectrum: *m*/*z* 361 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 66.50; H 3.22; N 7.69. $C_{20}H_{12}N_2OS_2$. Calculated, %: C 66.64; H 3.36; N 7.77.

3-(1*H*-Indol-3-yl)-2-(4-phenyl-1,3-thiazol-2-yl)-prop-2-enenitrile (XIIId). Yield 2.4 g (73%), yellow crystals, mp 256–258°C (from BuOH). IR spectrum, ν, cm^{-1}: 3300 (NH), 2215 (C≡N). ^1H NMR spectrum, δ, ppm: 7.24–7.33 m (2H, H_{arom}), 7.40 t (1H, H_{arom}, J = 7.9 Hz), 7.50 t (2H, H_{arom}, J = 7.5 Hz), 7.58 d (1H, H_{arom}, J = 7.5 Hz), 8.01 d (3H, H_{arom}, J = 7.0 Hz), 8.16 s (1H, 5″-H), 8.51d (1H, H_{arom}, J = 3.0 Hz), 8.55 s (1H, CH=), 12.35 br.s (1H, NH). Mass spectrum: *m*/*z* 328 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 73.20; H 3.84; N 12.72. $C_{20}H_{13}N_3S$. Calculated, %: C 73.37; H 4.00; N 12.83.

3-(4-Dimethylaminophenyl)-2-(4,5,6,7-tetrahydro-1,3-benzothiazol-2-yl)prop-2-enenitrile (XIV) was synthesized in a similar way from 2.3 g (10 mmol) of 2-cyano-3-(4-dimethylaminophenyl)prop-2-enethioamide (**IXb**) and 1.8 g (10 mmol) of 2-bromocyclohexan-1-one (**XII**). Yield 2.1 g (69%), yellow crystals, mp 68–70°C (from PrOH). IR spectrum: ν 2216 cm^{-1} (C≡N). ^1H NMR spectrum, δ, ppm: 1.81–1.92 m (4H, CH_2), 2.65–2.71 m (2H, CH_2), 2.75–2.81 m (2H, CH_2), 3.08 s (6H, Me), 6.71 d and 7.79 d (2H each, H_{arom}, J = 8.1 Hz), 7.81 s (1H, CH=). Mass spectrum: *m*/*z* 310 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 69.75; H 6.11; N 13.42. $C_{18}H_{19}N_3S$. Calculated, %: C 69.87; H 6.19; N 13.58.

5-[(1*H*-Indol-3-yl)methylidene]-2,2-dimethyl-1,3-dioxane-4,6-dione (XVI) was synthesized as described above for compounds **IXa**–**IXe** from 1.9 g of (10 mmol) dinitrile **VIIIe** and 1.44 g (10 mmol) of Meldrum's acid (**XV**). Yield 1.9 g (70%), colorless crystals, mp 234–235°C (from EtOH). IR spectrum, ν, cm^{-1}: 3325 (NH), 1714 (C=O). ^1H NMR spectrum, δ, ppm: 1.72 s (6H, Me), 7.32–7.37 m (2H, H_{arom}), 7.62 d (1H, H_{arom}, J = 6.4 Hz), 7.91 d (1H, H_{arom}, J = 5.9 Hz), 8.75 s (1H, CH=), 9.34 s (1H, 2′-H), 12.90 br.s (1H, NH). Mass spectrum: *m*/*z* 270 (I_{rel} 100%) $[M - 1]^+$. Found, %: C 66.35; H 4.72; N 5.09. $C_{15}H_{13}NO_4$. Calculated, %: C 66.42; H 4.83; N 5.16.

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