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Synthesis of 2-[4-Aryl(hetaryl, cyclopropyl)thiazol-2-yl]-3-hetarylacrylonitriles by Recyclizations of 2,6-Diamino-4-hetaryl-4*H*-thiopyran-3,5-dicarbonitriles with α-Bromoketones

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Abstract—Recyclization of 2,6-diamino-4-hetaryl-4*H*-thiopyran-3,5-dicarbonitriles with α-bromoketones afforded 2-[4-aryl(hetaryl, cyclopropyl)thiazol-2-yl]-3-hetarylacrylonitriles.

Keywords: 4*H*-thiopyranes, α-bromoketone, 3-hetaryl-2-(thiazol-2-yl)acrylonitriles, recyclization

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2,6-Diamino-4-aryl(hetaryl)-3,5-dicyano-4*H*-thiopyrans synthesized by reacting aryl(hetaryl)methylidenemalononitriles with cyanothioacetamide are known to transform into 6-amino-4-aryl(hetaryl)-3,5-dicyanopyridine-2(1*H*)-thiones when refluxing in ethanol in the presence of amine [1–4]. In an aqueous ethanol this reaction resulted in the formation of 4-aryl-6-hydroxy-3,5-dicyanopyridine-2(1*H*)-thiones [5–7]. Cross-recyclization of these thiopyrans afforded 4-aryl-2,6-dihydrazino-3,5-dicyanopyridines (in the presence of twofold excess of hydrazine) [8], substituted 3-(1-pyridinium)-5-cyano-3,4-*trans*-1,2,3,4-tetrahydropyridine-6-thiolates (in the presence of an equimolar amount of pyridinium ylides) [9], substituted 2-amino-4-aryl-7,7-dimethyl-5-oxo-3-cyano-5,6,7,8-tetrahydro-4*H*-benzo[*b*]-pyrans (in the presence of dimedone) [10], and 4-aryl-5,6-tri(tetra)methylene-3-cyanopyridine-2(1*H*)-thiones (in the presence of cycloalkanones enamines) [11, 12].

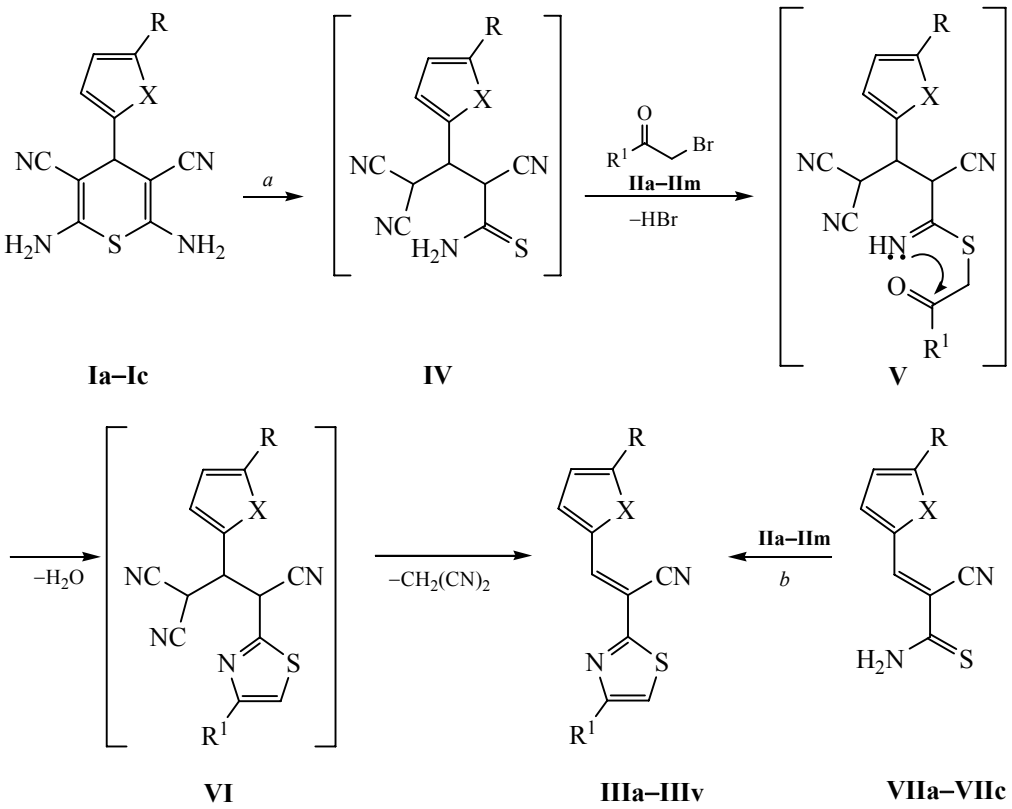
Further developing our previous studies on the chemistry 4*H*-thiopyrans [13–16], here we report on the recyclization of substituted 2,6-diamino-4-[2-furyl-(thienyl)]-4*H*-thiopyran-3,5-dicarbonitriles **Ia**–**Ic** with α-bromoketones **IIa**–**IIo** in boiling 2-propanol in the presence of triethylamine.

The products of this transformation were 2-[4-aryl-(hetaryl, cyclopropyl)thiazol-2-yl]-3-hetarylakrylonitriles **IIIa**–**IIIv** (Tables 1, 2).

The reaction probably comprises the thiopyran ring opening under the action of temperature and a catalyst to form the corresponding intermediates **IV**, which were regioselectively alkylated with α-bromoketones **IIa**–**IIo** to yield compounds **V**. The latter underwent intramolecular condensation to the Hantzsch thiazoles **VI** followed by malonodinitrile elimination and the formation of substituted acrylonitriles **IIIa**–**IIIv** (method *a*). This reaction pathway was proved by replacing α-bromo ketones **II** with alkyl halides without carbonyl function, which led to the retention of malonodinitrile moiety in the molecules of intermediates **V** and to pyridine ring formation [15] (Scheme 1).

The structures of compounds **IIIa**–**IIIv** were confirmed by IR and ^{1}H NMR spectroscopy data (Table 2) as well as by authentic Hantzsch synthesis starting from acrylic acid thioamides **VIIa**–**VIIc** and α-bromo ketones **IIa**–**IIo** (method *b*). For example, in the IR spectra of acrylonitriles **IIIa**–**IIIv** there were characteristic absorption bands of the stretching vibrations of the conjugated cyano group in the range of 2202–2229 cm^{-1}. ^{1}H NMR spectra contained the signals of the vinyl moiety and H^5 of the thiazole ring, which is typical for this class of compounds [17] as well as the proton signals of aromatic substituents. The value of the molecular ion peak in the mass spectrum

Scheme 1.



Ia–Ic **IV** **V** **VI** **IIIa–IIIv** **VIIa–VIIc**

I, **VII**, R = H, X = O (**a**), Me, O (**b**), H, S (**c**); **II**, R^1 = cyclopropyl (**a**), 6,8-dibromocoumarin-3-yl (**b**), 4-BuC_6H_4 (**c**); 4-PhC_6H_4 (**d**), coumarin-3-yl (**e**), 4-MeC_6H_4 (**f**), 3,4-$Cl_2C_6H_3$ (**g**), 2-HOC_6H_4 (**h**), Ph (**i**), 2,4-$Me_2C_6H_3$ (**j**), 4-ClC_6H_4 (**k**), 4-BrC_6H_4 (**l**), 4-$MeOC_6H_4$ (**m**); **III**, R = H, X = O, R^1 = cyclopropyl (**a**), 6,8-dibromocoumarin-3-yl (**b**), 4-BuC_6H_4 (**c**), 4-PhC_6H_4 (**d**), coumarin-3-yl (**e**), 4-MeC_6H_4 (**f**), 3,4-$Cl_2C_6H_3$ (**g**), 2-HOC_6H_4 (**h**); R = Me, X = O, R^1 = 4-BuC_6H_4 (**i**), Ph (**j**), cyclopropyl (**k**), 4-PhC_6H_4 (**l**), 2,4-$Me_2C_6H_3$ (**m**), coumarin-3-yl (**n**), 4-MeC_6H_4 (**o**), 3,4-$Cl_2C_6H_3$ (**p**), 4-ClC_6H_4 (**q**), 4-BrC_6H_4 (**r**); R = H, X = S, R^1 = cyclopropyl (**s**), 4-PhC_6H_4 (**t**), coumarin-3-yl (**u**), 4-$MeOC_6H_4$ (**v**).

of compound **IIIh** corresponded to the "nitrogen rule" [18], and the existence of the ion $[M + 2]^+$ indicated the presence of the sulfur atom in the molecule [19].

When comparing the yields of the compounds III prepared according to the methods *a* and *b* it is seen (Table 1) that in the first case they are smaller obviously due to occurrence of the side reactions. The method *a* possesses a special synthetic importance because in some cases the aryl(hetaryl)methylidenecyanothioacetamides, which are necessary for the Hantzsch method implementing, failed to be isolated because of their easy dimerization [20], and alkylmethylidenecyanothioacetamides are hitherto unknown. Note that the organic compounds containing thiazole moiety are of special interest due to their wide occurrence in nature [21]. In particular, 4-(3-coumarinyl)thiazoles are known as phosphors [22], and 5-arylthiazoles show fungicidal [23], antiviral [24, 25] and anti-enzymatic [26, 27] activity.

EXPERIMENTAL

IR spectra were recorded on a X-29 spectrometer (from mulls in mineral oil). ^{1}H NMR spectra of the DMSO-d_6 or $CDCl_3$ solutions were registered on a Bruker WP-100SY (100 MHz, **IIIa**, **IIIc**–**IIIf**, **IIIk**–**IIIu**), Gemini-200 (200 MHz, **IIIv**), Bruker AM-300 (300 MHz, **IIIb**), Varian Mercury-400 (400 MHz, **IIIi**, **IIIj**), Bruker DR-500 (500 MHz, **IIIh**), and Bruker AM-300 (300 MHz, **IIIg**) spectrometers, internal reference TMS. Mass spectra were taken on a Kratos MS-890 spectrometer (EI, 70 eV) with a direct input of the sample into the ion source. Melting points were determined on a Koeffler heating block. The reaction

Table 1. Yields, melting points, and elemental analysis data of compounds **IIIa**–**IIIv**

Comp. no.	Yield, %, method *a*/*b*	mp, °C	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
IIIa	61/75	82	64.32	4.08	11.47	$C_{13}H_{10}N_2OS$	64.44	4.16	11.56
IIIb	68/74	218–220	45.01	1.42	5.45	$C_{19}H_8Br_2N_2O_3S$	45.27	1.60	5.56
IIIc	70/81	70–72	71.72	5.31	8.25	$C_{20}H_{18}N_2OS$	71.83	5.43	8.38
IIId	75/88	169–171	74.33	4.11	7.75	$C_{22}H_{14}N_2OS$	74.55	3.98	7.90
IIIe	69/76	219–221	65.77	3.04	7.96	$C_{19}H_{10}N_2O_3S$	65.89	2.91	8.09
IIIf	65/70	139–141	69.72	3.99	9.41	$C_{17}H_{12}N_2OS$	69.84	4.14	9.58
IIIg	78/90	170–172	55.20	2.11	7.89	$C_{16}H_8Cl_2N_2OS$	55.35	2.32	8.07
IIIh	60/72	169–170	65.14	3.31	9.37	$C_{16}H_{10}N_2O_2S$	65.29	3.42	9.52
IIIi	73/88	91–93	72.24	5.66	7.95	$C_{21}H_{20}N_2OS$	72.38	5.79	8.04
IIIj	64/78	128–130	69.70	3.96	9.42	$C_{17}H_{12}N_2OS$	69.84	4.14	9.58
IIIk	61/70	70–71	65.57	4.62	10.80	$C_{14}H_{12}N_2OS$	65.60	4.72	10.93
IIIl	85/91	161–163	75.09	4.22	7.48	$C_{23}H_{16}N_2OS$	74.98	4.38	7.60
IIIm	62/80	110–112	71.06	4.91	8.65	$C_{19}H_{16}N_2OS$	71.22	5.03	8.74
IIIn	73/84	173–175	66.48	3.19	7.58	$C_{20}H_{12}N_2O_3S$	66.66	3.36	7.77
IIIo	69/75	138–140	70.42	4.55	8.97	$C_{18}H_{14}N_2OS$	70.56	4.61	9.14
IIIp	61/77	145–147	56.39	2.84	7.66	$C_{17}H_{10}Cl_2N_2OS$	56.52	2.79	7.75
IIIq	72/84	141–143	62.37	3.22	8.42	$C_{17}H_{11}ClN_2OS$	62.48	3.39	8.57
IIIr	65/80	133–135	54.86	3.07	7.38	$C_{17}H_{11}BrN_2OS$	55.00	2.99	7.55
IIIs	79/80	105–107	60.29	3.77	10.71	$C_{13}H_{10}N_2S_2$	60.44	3.90	10.84
IIIt	84/93	190–191	71.28	3.66	7.43	$C_{22}H_{14}N_2S_2$	71.32	3.81	7.56
IIIu	66/72	215–217	62.86	2.59	7.64	$C_{19}H_{10}N_2O_2S_2$	62.97	2.78	7.73
IIIv	64/69	153–155	62.81	3.62	8.54	$C_{17}H_{12}N_2OS_2$	62.94	3.73	8.63

Table 2. Parameters of IR and ^{1}H NMR spectra of compounds **IIIa**–**IIIv**

Comp. no.	ν(C≡N, C=O), cm^{-1}	δ_H, ppm (*J*, Hz)	
		CH= (1H, s), $H^5_{thiazole}$ (1H, s)	Other signals
IIIa	2211	7.96, 7.38	0.78–1.06 m (4H, $2CH_2$), 2.11 m (1H, CH), 6.74 d.d (1H, H^4, furan, *J* 2.4, *J* 3.1), 7.31 d (1H, H^3, furan, *J* 2.9), 8.09 d (1H, H^5, furan, *J* 1.2)
IIIb	2215, 1718	8.12, 8.05	6.79 d.d (1H, H^4, furan, *J* 2.9, *J* 2.9), 7.32 d (1H, H^3, furan, *J* 2.9), 7.99 d (1H, H^5, furan, *J* 1.2), 8.01 s (1H, H^7, coumarin), 8.48 s (1H, H^5, coumarin), 8.81 s (1H, H^4, coumarin)
IIIc	2229	8.16, 8.07	0.89 t (3H, CH_3, *J* 7.2), 1.11–1.72 m (4H, $2CH_2$), 2.60 t (2H, CH_2, *J* 6.0), 6.83 d.d (1H, H^4, furan, *J* 2.4, *J* 2.9), 7.37 d (1H, H^3, furan, *J* 2.9), 7.26 d (2H, H_{Ar}, *J* 7.0), 7.91 d (2H, H_{Ar}, *J* 7.0), 8.00 d (1H, H^5, furan, *J* 1.2)
IIId	2220	8.27, 8.15	6.82 d.d (1H, H^4, furan, *J* 2.4, *J* 3.2), 7.31 d (1H, H^3, furan, *J* 2.9), 7.35–7.49 m (3H, H_{Ar}), 7.55–7.72 m (2H, H_{Ar}), 7.77 d (2H, H_{Ar}, *J* 7.1), 8.01 d (2H, H_{Ar}, *J* 7.1), 8.07 d (1H, H^5, furan, *J* 1.2)
IIIe	2219, 1722	8.44, 8.14	6.81 d.d (1H, H^4, furan, *J* 2.4, *J* 3.0), 7.21–7.47 m (3H, $H^{6,7}$, coumarin; H^3, furan), 7.62 d (1H, H^8, coumarin, *J* 8.0), 7.86 d (1H, H^5, coumarin, *J* 8.0), 8.01 d (1H, H^5, furan, *J* 1.2), 8.77 s (1H, H^4, coumarin)

Table 2. (Contd.)

Comp. no.	ν(C≡N, C=O), cm^{-1}	δ_H, ppm (*J*, Hz)	
		CH= (1H, s), $H^5_{thiazole}$ (1H, s)	Other signals
IIIf	2202	8.23, 8.12	2.4 s (3H, CH_3), 6.83 d.d (1H, H^4, furan, *J* 2.4, *J* 3.1), 7.33 d (1H, H^3, furan, *J* 2.8), 7.28 d (2H, H_{Ar}, *J* 7.7), 7.91 d (2H, H_{Ar}, *J* 7.7), 8.01 d (1H, H^5, furan, *J* 1.3)
IIIg	2215	8.06, 8.02	7.24 d.d (1H, H^4, furan, *J* 2.4, *J* 2.4), 7.55 s (1H, H_{Ar}), 7.69 d (1H, H^3, furan, *J* 2.8), 7.72 d (1H, H_{Ar}, *J* 7.4), 7.76 d (1H, H_{Ar}, *J* 7.4), 7.91 d (1H, H^5, furan, *J* 1.3)
IIIh	3606 (OH), 2227	8.18, 7.93	6.70 d.d (1H, H^4, furan, *J* 1.8, *J* 2.0), 6.83–6.96 m (3H, H_{Ar}), 7.15 t (1H, H_{Ar}, *J* 7.0), 7.32 d (1H, H^3, furan, *J* 3.6), 8.07 d (1H, H^5, furan, *J* 1.3), 10.37 br. s (1H, OH)
IIIi	2216	8.11, 8.04	0.88 t (3H, CH_3, *J* 7.3), 1.14–1.72 m (4H, $2CH_2$), 2.41 s (3H, CH_3), 2.58 t (2H, CH_2, *J* 7.1), 6.46 d (1H, H^3, furan, *J* 2.9), 7.24 d (1H, H^4, furan, *J* 2.9), 7.28 d (2H, H_{Ar}, *J* 7.1), 7.89 d (2H, H_{Ar}, *J* 7.1)
IIIj	2220	8.22, 8.07	2.43 s (3H, CH_3), 6.49 d (1H, H^4, furan, *J* 2.9), 7.28 d (1H, H^3, furan, *J* 2.9), 7.39 t (1H, H_{Ar}, *J* 6.9), 7.47 t (2H, H_{Ar}, *J* 6.9), 8.02 d (2H, H_{Ar}, *J* 7.0)
IIIk	2219	7.87, 7.36	0.81–1.07 m (4H, $2CH_2$), 1.99–2.27 m (1H, CH), 2.42 s (3H, CH_3), 6.47 d (1H, H^4, furan, *J* 2.9), 7.24 d (1H, H^3, furan, *J* 2.9)
IIIl	2203	8.25, 8.00	2.44 s (3H, CH_3), 6.51 d (1H, H^4, furan, *J* 3.0), 7.31 d (1H, H^3, furan, *J* 3.0), 7.41–7.62 m (2H, H_{Ar}), 7.68–7.89 m (5H, H_{Ar}), 8.08 d (2H, H_{Ar}, *J* 7.6)
IIIm	2214	7.98, 7.79	2.31 s (3H, CH_3), 2.42 s (6H, $2CH_3$), 6.48 d (1H, H^4, furan, *J* 3.0), 7.08 d (1H, H_{Ar}, *J* 8.5), 7.12 s (1H, H_{Ar}), 7.26 d (1H, H^3, furan, *J* 3.0), 7.54 d (1H, H_{Ar}, *J* 8.5)
IIIn	2202, 1719	8.44, 8.06	2.44 s (3H, CH_3), 6.52 d (1H, H^4, furan, *J* 3.0), 7.26 d (1H, H^3, furan, *J* 3.0), 7.38 t (1H, H^7, coumarin, *J* 8.0), 7.43 d (1H, H^5, coumarin, *J* 8.0), 7.24 t (1H, H^6, coumarin, *J* 8.0), 7.91 d (1H, H^8, coumarin, *J* 8.0), 8.81 s (1H, H^4, coumarin)
IIIo	2211	8.10, 8.03	2.34 s (3H, CH_3), 2.42 s (3H, CH_3), 6.48 d (1H, H^4, furan, *J* 3.0), 7.19 d (1H, H^3, furan, *J* 3.0), 7.24 d (2H, H_{Ar}, *J* 7.7), 7.89 d (2H, H_{Ar}, *J* 7.7)
IIIp	2218	8.24, 8.15	6.49 d (1H, H^4, furan, *J* 3.0), 2.45 s (3H, CH_3), 7.32 d (1H, H^3, furan, *J* 3.0), 7.67 d (1H, H_{Ar}, *J* 8.4), 7.98 d (1H, H_{Ar}, *J* 8.4), 8.10 s (1H, H_{Ar})
IIIq	2209	8.24, 8.01	2.43 s (3H, CH_3), 6.52 d (1H, H^4, furan, *J* 3.1), 7.28 d (1H, H^3, furan, *J* 3.1), 7.51 d (2H, H_{Ar}, *J* 8.6), 7.98 d (2H, H_{Ar}, *J* 8.6)
IIIr	2214	8.26, 8.05	2.43 s (3H, CH_3), 6.51 d (1H, H^4, furan, *J* 2.9), 7.29 d (1H, H^3, furan, *J* 2.9), 7.65 d (2H, H_{Ar}, *J* 7.5), 7.95 d (2H, H_{Ar}, *J* 7.5)
IIIs	2228	8.41, 7.38	0.85–1.07 m (4H, $2CH_2$), 1.99–2.34 m (1H, CH), 7.29 d.d (1H, H^4, thiophene, *J* 6.1, *J* 4.5), 7.93 d (1H, H^3, thiophene, *J* 4.6), 8.03 d (1H, H^5, thiophene, *J* 3.7)
IIIt	2226	8.64, 8.33	6.92 d (2H, H_{Ar}, *J* 8.4), 7.28–7.54 m (4H, H_{Ar}), 7.69–8.21 m (6H, H_{Ar})
IIIu	2221, 1712	8.51, 8.47	7.22–7.48 m (2H, H_{Ar}), 7.59–7.66 m (1H, H_{Ar}), 7.78 m (3H, H_{Ar}), 8.04 d (1H, H^5, thiophene, *J* 3.8), 8.74 s (1H, H^4, coumarin)
IIIv	2217	8.44, 8.15	3.84 s (3H, CH_3), 7.17 d (2H, H_{Ar}, *J* 8.0), 7.32 d.d (1H, H^4, thiophene, *J* 5.8, *J* 3.4), 7.91 d (1H, H^3, thiophene, *J* 4.5), 8.04 d (2H, H_{Ar}, *J* 8.0), 8.22 d (1H, H^5, thiophene, *J* 3.7)

progress and the purity of the obtained compounds were monitored by TLC on Silufol UV-254 plates, eluting with acetone–hexane (3:5) and detecting with iodine vapor or UV irradiation.

2,6-Diamino-4-hetaryl-4*H*-thiopyran-3,5-dicarbonitriles **Ia**–**Ic** and 3-hetarylmethylidenecyanothioacetamides **VIIa**–**VIIc** were prepared by the proce-dures reported in [28] and [29], respectively.

2-[4-Aryl(hetaryl, cyclopropyl)thiazol-2-yl]-3-hetarylacrylonitriles (IIIa–IIIv). *a*. A mixture of 10 mmol of thiopyran **I**, 10 mmol of α-bromoketone **II**, and 1.4 mL (10 mmol) of triethylamine in 25 mL of

2-propanol was refluxed for 8 h and filtered. After 48 h the precipitate was separated, washed with 2-propanol and hexane, and recrystallized from butanol.

b. A mixture of 10 mmol of acrylic acid thioamide **VII** and 10 mmol of α-bromoketone **II** in 25 mL of DMF was stirred at 20°C for 30 min and then left standing for 24 h. The reaction mixture was diluted with an equal volume of water, and the resulting precipitate was filtered off. The melting points and R_f values of compounds **IIIa**–**IIIv** obtained were identical to those of the samples synthesized by the method *a*.

Mass spectrum of compound **IIIh**, *m*/*z* (I_{rel}, %): 296 $[M + 2]^+$ (4), 295 $[M + 1]^+$ (16), 293 $[M - 1]^+$ (15), 266 (14), 253 (95), 240 (31), 121 (75), 89 (27), 77 (22), 63 (20), 51 (14), 39 (13).

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