Chemistry of Heterocyclic Compounds, Vol. 32, No. 1, 1996

NEW APPROACHES TO THE SYNTHESIS OF FUNCTIONALLY SUBSTITUTED PYRIDO[3',2':4,5]THIENO[3,2-*b*]PYRIDINES AND THE STRUCTURE OF THE PRODUCTS OBTAINED

V. L. Ivanov, V. A. Artemov, L. A. Rodinovskaya, A. M. Shestopalov, V. N. Nesterov, Yu. T. Struchkov,* and V. P. Litvinov

Substituted pyrido[3',2':4,5]thieno[3,2-b]pyridines were obtained by the reaction of 3-amino-2-benzoylthieno[2,3-b]pyridines with malononitrile and the reaction of 3-cyanopyridine-2(1H)-thiones with 2-aryl-3-bromo-1,1-dicyanopropene. 2-Amino-4-(4-bromophenyl)-7,9-dimethyl-3-cyanopyrido[3',2':4,5]thieno[3,2-b]pyridine was used for the synthesis of a derivative of pyrido[3",2":4',5']thieno[2',3':5,6]pyrido[2,3-d]pyrimidine. The structure of these compounds was confirmed by spectral data and x-ray diffraction structural analysis.

Pyrido[3',2':4,5-thieno- and pyrido[3',2':4,5]selenopheno[2,3-*d*]pyrimidines were obtained in our laboratory by the reaction of 3-cyanopyridine-2(1H)-thiones and 3-cyanopyridine-2(1H)-selenones with N-cyanochloracetoamide [1, 2]. The formation of these condensed pyrimidines was found to proceed as the result of a sequence of consecutive reactions: nucleophilic substitution at the sulfur atom, Thorpe–Ziegler closure of the thiophene ring, and Thorpe–Guareschi closure of the pyridine ring. This may be considered a multistep reaction cascade.

In the present work, we are the first to report the use of cascade reactions in the synthesis of pyrido[3',2':4,5]-thieno[3,2-*b*]pyridines. Only two approaches have been offered for the synthesis of such compounds. The first method involves the formation of the pyridine ring by the reaction of 3-aminothieno[2,3-*b*]pyridine with the acetal of malonodialdehyde [3]. In the second method, the reaction of 2-acyl-3-aminothieno[2,3-*d*]pyridine with ethyl orthoformate or the diethylacetal of dimethylformamide is used [4]. Both these methods are carried out under vigorous conditions, which leads to difficulties in isolating the desired products and their low yields. Furthermore, the starting compounds used are not readily available.

We have developed new pathways for the synthesis of functionally-substituted pyrido[3',2':4,5]thieno[3,2-*b*]pyridines (Ia-Ih) based on the use of 3-cyanopyridine-2(1H)-thiones (IIa-IIg), which permits a more facile construction of these tricyclic systems. The first such method involves the reaction of the products of the condensation of thiones IIa and IIb and phenacyl bromide, namely, 3-amino-2-benzoylthieno[2,3-*b*]pyridines (IIIa and IIIb) with malononitrile under the Friedlander reaction conditions (method A, scheme I). This reaction proceeds upon heating in pyridine and leads to the desired products in good yield. However, in light of the vigorous conditions, there is significant tar formation, which complicates the isolation and purification of the desired pyridothienopyridine. The results obtained by this method were partially presented at the Ukrainian Conference on Organic Chemistry [5].

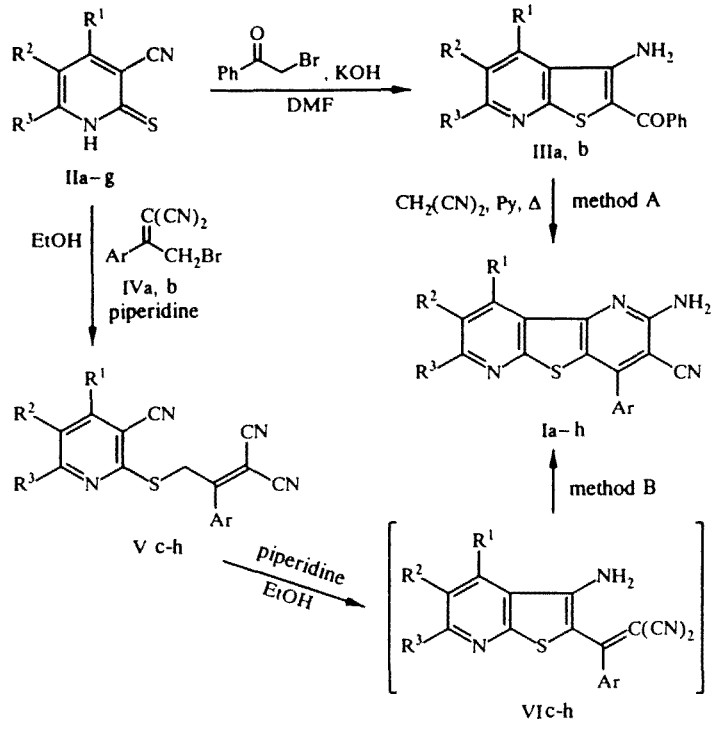
The second synthetic method for condensed products I involves the reaction of pyridinethiones IIc-IIg with 2-bromo-1-arylethylidenemalononitrile (IVa and IVb) (method B, scheme 1). Reagents IV are blocks containing fragments of an aryl bromomethyl ketone and malononitrile. The cascade reactions in method B proceed under much milder conditions and the yields of pyridothienopyridines I are much higher. Thus, for example, heating pyridinethione IIc with reagents IVa and IVb

*Deceased.

N. D. Zelinskii Institute of Organic Chemistry, Russian Academy of Sciences, 117913, Moscow. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 1, pp. 115-122, January, 1996. Original article submitted May 20, 1995.

0009-3122/96/3201-0105$15.00

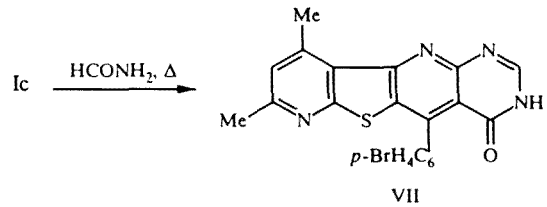
Scheme 1



I—III, V, VI a $R^1 = R^2 = H$, $R^3 = Ph$; b $R^1 = CH_3$, $R^2 = H$, $R^3 = Ph$;
c $R^1 = R^3 = CH_3$, $R^2 = H$; d $R^1 = R^2 = H$, $R^3 = CH_3$; e $R^1 = R^3 = Ph$, $R^2 = H$; f $R^1 = H$, $R^2R^3 = (CH_2)_3$;
g $R^1 = H$, $R^2 = R^3 = (CH_2)_4$; h $R^1 = R^3 = CH_3$, $R^2 = H$
Ia,b,h IVa, Vh, VIh, Ar = Ph; I c-g , IVb, Vc-g, VIc-g Ar = 4-BrC_6H_4

The mechanism for this transformation involves the initial regioselective alkylation of pyridinethiones II at the sulfur atom with formation of substituted pyridines V. When an insufficient amount of base is used in the case of 4,6-dimethyl derivatives IIc and IIh, the reaction mixture yields intermediates Vc and Vh, respectively. The next step features Thorpe–Ziegler intramolecular cyclization of V involving the acidic methylene group of the side-chain and 3-CN group, which probably leads to thienopyridines VI. It proved impossible to isolate intermediates VI, possibly due to the very rapid cyclization of these species to give a pyridine ring. Indeed, such reactions of amino and nitrile groups proceed very readily [6]. Thus, the cascade reactions leading to the formation of pyridothienopyridines involve consecutive alkylation, Thorpe–Ziegler cyclization, and intramolecular reaction of the amino and nitrile groups. The yields and physical indices of products I are given in Table 1.

Scheme 2



VII

Products I undergo reactions characteristic for pyridines containing an enaminonitrile fragment. The reaction of pyridothienopyridine Ic with formamide leads to formation of a pyrimidine ring in tetracyclic product VII (scheme 2).

The IR spectra of these products provided sufficient information for determination of their structure. Thus, the spectra of V show two nitrile group bands: at 2240 (3-CN) and 2215 cm^{-1} (side-chain CN). These spectra lack bands for the amino group. The bands for the 3-CN group at 2240 cm^{-1} disappear upon the cyclization of Vc and Vh and the spectra of products

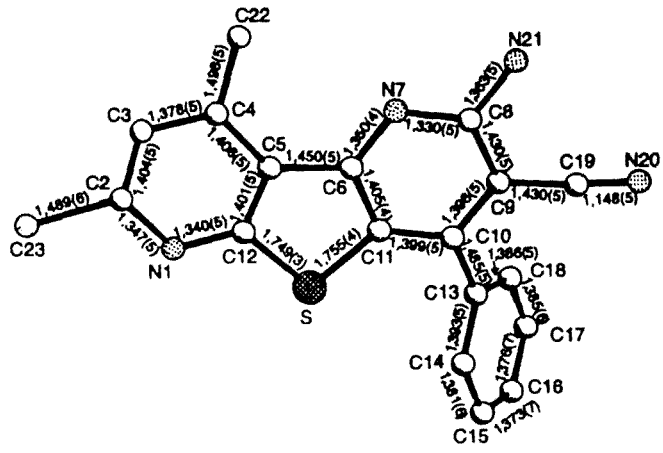


Fig. 1. General view of Ih and the bond lengths in this molecule.

Ic and Ih show characteristic amino group bands (see Experimental and Table 1). However, these results do not indicate unequivocally whether these compounds have structure I or VI. Thus, we carried out an x-ray diffraction structural analysis of Ih, which showed that it is a tricyclic system (see below).

The chemical shifts of the amino group protons in I are also characteristic and are seen usually as rather narrow singlets in the vicinity of 7 ppm. An exception is found for 9-phenyl derivative Ie, in which the amino group protons are probably strongly shielded by the ring current of the phenyl substituent. Indeed, the x-ray diffraction structural analysis data for Ih indicate contact of $N_{(1)}$ and substituent R^1. This contact is also seen in the chemical shifts of the methyl substituents in the PMR spectra of Ic and Ih. Thus, the signals of the 9-CH_3 (R^1) group protons are found at much lower field than 7-CH_3. This same effect has been described for the pyrido[3′,2′:4,5]thieno[3,2-*d*]pyrimidine system [2].

Our x-ray diffraction structural analysis unequivocally proved the structure of Ih. Figure 1 shows a general view of this molecule and its bond lengths. The bond angles are given in Table 2.

The bond lengths in pyridothienopyridine Ih have the usual values [7]. Its tricyclic system is planar to ±0.020 Å. The short nonbonding contacts $S\cdots C_{(14)}$ (3.230(4) Å) and $C_{(18)}\cdots C_{(19)}$ (3.127(5) Å) (the sum of the van der Waals radii for sulfur and carbon is 3.50 Å and double the carbon radius is 3.40 Å [8]) cause a twist in the phenyl substituent relative to the tricyclic system by 55.2°. A shortened intramolecular contact is found in this compound $C_{(22)}\cdots N_{(7)}$ (3.053 Å, the sum of the van der Waals radii of the CH_3 and NH_2 groups is 3.25 Å [8]). Conjugation between the NH_2 group and pyridine ring causes marked shortening of the $C_{(8)}-N_{(21)}$ bond to 1.363 Å, which is comparable to the length of the C–N bond in both pyridine rings in this molecule (Fig. 1).

A pair of weak intramolecular N–H···N hydrogen bonds (N···N 3.107(5), N–H 0.86(5), H···N 2.42(5) Å, N–H···N 138(5)°) links molecules of Ih in the crystal to give asymmetrical dimers (Fig. 2). Analysis of the molecular packing in the crystal shows that there are no other shortened intramolecular contacts.

EXPERIMENTAL

X-Ray Diffraction Structural Analysis of Id. The unit cell parameters for orthorhombic crystals of Id at 20°C are as follows: a = 35.622(2), b = 10.935(3), c = 8.2282(4) Å, V = 3226(3) Å^3, d_{calc} = 1.367 g/cm^3, Z = 8, space group PbCn. The unit cell parameters and the intensities of 2652 independent reflections were measured on a Siemens P3/PC four-circle automatic diffractometer using λMoKα radiation, graphite monochromator, and $\theta/2\theta$ scanning to θ_{max} = 28°. The structure was solved by the direct method, which revealed all the nonhydrogen atoms and was refined by the full-matrix method of least squares anisotropically for the nonhydrogen atoms using 2267 reflections with $I > 3\sigma(I)$. All the hydrogen atoms were found in the difference map and refined isotropically. The final R = 0.069 (R_w = 0.064). All the calculations were carried out using the SHELXTL PLUS program (PC version). The atomic coordinates are given in Table 2. The temperature factors may be obtained from the authors.

TABLE 1. Yields and Physical Indices of Pyridothienopyridines Ia-Ih

Compound	Chemical formula	mp, °C	Elemental analysis data found, %/calculated, %			IR spectrum, ν, cm^{-1}	^1H NMR spectrum, δ, ppm, coupling constants (J), Hz					Yield, %
			C	H	N		R^1	R^2	R^3	NH_2 s, 2H	Ar	
Ia	$C_{23}H_{14}N_4S$	273...274	60,29 60,44	5,43 5,46	21,72 21,69	3414, 3318, 3215 (NH); 2219 (CN), 1638	8,50 d, J = 8 (1H)	8,05 d, J = 8 (1H)	7,50...8,00* m (10H)	7,06	*	75
Ib	$C_{24}H_{16}N_4S$	255...256	73,55 73,45	4,07 4,11	14,30 14,28	3410, 3327, 3225 (NH); 2220 (CN); 1640	2,88 s (3H)	7,94 s (1H)	7,34...8,20 m (10H)*	6,97	*	67
Ic	$C_{19}H_{13}BrN_4S$	319...320	56,01 55,76	3,11 3,20	13,60 13,69	3484, 3368 (NH); 2225 (CN); 1627, 1548, 1490	2,94 s (3H)	7,24 s (3H)	2,57 s (3H)	7,00	7,64 d (2H), 7,85 d (2H)	95
Id	$C_{18}H_{11}BrN_4S$	254...256	54,74 54,70	2,75 2,80	14,20 14,17	3485, 3380 (NH); 2220 (CN); 1640, 1531, 1492	8,36 d, J = 8 (1H)	7,43 d, J = 8 (1H)	2,62 s (3H)	7,13	7,65 d (2H), 7,85 d (2H)	72
Ie	$C_{29}H_{17}BrN_4S$	271...272	65,31 65,30	3,33 3,21	10,42 10,50	3425, 3304, 3208 (NH); 2222 (CN); 1632, 1568, 1522, 1500	8,12...8,18 m, o-H(2H); 7,00...7,60 m, $м$-H and p-H (3H)	8,02 s (1H)	7,50...7,60 $м,o$-H (2H), 7,00...7,60 $м$,p- and $м$-H (3H)	7,95	7,67, d (2H), 7,86 d (2H)	57
If	$C_{20}H_{13}BrN_4S$	305...307	56,88 57,02	3,07 3,11	13,31 13,30	3480, 3380, 3350 (NH); 2220 (CN); 1616, 1548, 1480	8,28 s (1H)	2,13 t (2H), 3,07 d (4H)		7,11	7,67 d (2H), 7,86 d (2H)	84
Ig	$C_{21}H_{15}BrN_4S$	308...310	57,99 57,94	3,47 3,47	12,80 12,87	3484, 3360 (NH); 2222 (CN); 1615, 1546, 1492	8,18 s (1H)	1,75...1,95 m (4H), 2,90...3,05 m (4H)		7,12	7,67 d (2H), 7,86 d (2H)	87
Ih	$C_{19}H_{14}N_4S$	289...290	68,94 69,07	4,11 4,27	17,02 16,96	3517, 3408 (NH); 2218 (CN); 1620, 1505, 1438	2,94 s (3H)	7,22 s (1H)	2,55 s (3H)	6,98	7,58...7,70 m (5H)	94

*Proton signals overlap.

TABLE 2. Bond Angles ω in Ih

Bond angle	ω, deg	Bond angle	ω, deg
C(11)—S—C(12)	90,9(2)	C(8)—C(9)—C(19)	117,2(3)
C(2)—N(1)—C(12)	115,4(3)	C(10)—C(9)—C(19)	122,2(3)
C(6)—N(7)—C(8)	116,9(3)	C(9)—C(10)—C(11)	115,6(3)
N(1)—C(2)—C(3)	122,0(3)	C(9)—C(10)—C(13)	123,0(3)
N(1)—C(2)—C(23)	116,5(4)	C(11)—C(10)—C(13)	121,3(3)
C(3)—C(2)—C(23)	121,5(4)	S—C(11)—C(6)	112,3(2)
C(2)—C(3)—C(4)	122,1(3)	S—C(11)—C(10)	127,1(2)
C(3)—C(4)—C(5)	116,7(3)	C(6)—C(11)—C(10)	120,5(3)
C(3)—C(4)—C(22)	120,7(3)	S—C(12)—N(1)	120,2(2)
C(5)—C(4)—C(22)	122,6(3)	S—C(12)—C(5)	113,0(3)
C(4)—C(5)—C(6)	131,3(3)	N(1)—C(12)—C(5)	126,7(3)
C(4)—C(5)—C(12)	117,1(3)	C(10)—C(13)—C(14)	121,0(3)
C(6)—C(5)—C(12)	111,6(3)	C(10)—C(13)—C(18)	119,9(3)
N(7)—C(6)—C(5)	124,3(3)	C(14)—C(13)—C(18)	119,0(3)
N(7)—C(6)—C(11)	123,5(3)	C(13)—C(14)—C(15)	120,5(4)
C(5)—C(6)—C(11)	112,2(3)	C(14)—C(15)—C(16)	119,9(4)
N(7)—C(8)—N(21)	116,2(3)	C(15)—C(16)—C(17)	120,1(4)
N(7)—C(8)—C(9)	122,9(3)	C(16)—C(17)—C(18)	120,4(4)
N(21)—C(8)—C(9)	120,9(3)	C(13)—C(18)—C(17)	120,0(4)
C(8)—C(9)—C(10)	120,6(3)	N(20)—C(19)—C(9)	174,6(4)

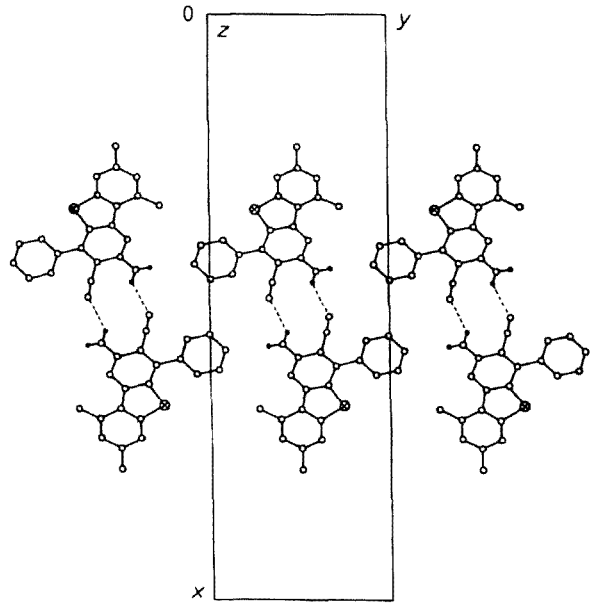


Fig. 2. Projection of the crystal structure of Ih onto the *ab* plane (the intermolecular N—H···N hydrogen bonds are indicated by dashed lines).

The melting points were determined on a Koefler block. The IR spectra were taken on a Specord M-80 spectrometer for KBr pellets. The ^{1}H NMR spectra were taken on a Bruker WM-250 spectrometer for DMSO-d_6 solutions. The ^{13}C NMR spectra were taken on a Bruker WM-300 spectrometer in DMSO-d_6 solutions. The mass spectra were taken on a Varian MAT CH-6 mass spectrometer at 70 eV. The elemental analyses for C, H, N were taken on a Perkin-Elmer C,H,N analyzer.

The elemental analysis data were in accord with the calculated results.

The physical indices of products I are given in Table 1. The starting compounds were obtained according to reported methods: IIa-IIg [9, 10], IIIa, IIIb [9], IVa, and IVb [11].

TABLE 3. Atomic Coordinates ($\times 10^4$ for N, C, $\times 10^3$ for H) in Ih

Atom	x	y	z
$N_{(1)}$	3329(1)	2573(1)	784(1)
$N_{(7)}$	2768(1)	3866(2)	-546(4)
$N_{(20)}$	3849(1)	5525(2)	2335(4)
$N_{(21)}$	4848(1)	3337(3)	4753(5)
$C_{(2)}$	4378(1)	5962(3)	3789(5)
$C_{(3)}$	2611(1)	4956(3)	-899(5)
$C_{(4)}$	2776(1)	6055(3)	-422(5)
$C_{(5)}$	3106(1)	6105(3)	449(5)
$C_{(6)}$	3274(1)	4976(3)	818(4)
$C_{(7)}$	3615(1)	4683(3)	1694(5)
$C_{(8)}$	4155(1)	5107(4)	3074(5)
$C_{(9)}$	4247(1)	3835(3)	3155(5)
$C_{(10)}$	4011(1)	2961(3)	2459(5)
$C_{(11)}$	3683(1)	3417(3)	1750(5)
$C_{(12)}$	3091(1)	3918(3)	279(5)
$C_{(13)}$	4108(1)	1642(3)	2373(4)
$C_{(14)}$	3853(1)	750(3)	2866(5)
$C_{(15)}$	3938(1)	-476(4)	2697(6)
$C_{(16)}$	4272(1)	-822(4)	2002(6)
$C_{(17)}$	4528(1)	50(4)	1530(7)
$C_{(18)}$	4448(1)	1282(4)	1714(6)
$C_{(19)}$	4579(1)	3506(3)	4019(5)
$C_{(22)}$	3272(1)	7303(3)	953(7)
$C_{(23)}$	2247(1)	4924(6)	-1779(7)
$H_{(21)a}$*	458(1)	574(4)	427(6)
$H_{(21)b}$*	434(1)	674(4)	367(5)
$H_{(3)}$	256(1)	678(3)	-81(4)
$H_{(14)}$	363(1)	96(3)	345(5)
$H_{(15)}$	375(1)	-106(4)	308(6)
$H_{(16)}$	432(1)	-162(4)	182(5)
$H_{(17)}$	475(1)	-21(3)	102(5)
$H_{(18)}$	463(1)	184(3)	120(5)
$H_{(22)a}$	353(2)	747(4)	54(6)
$H_{(22)b}$	313(1)	807(4)	49(6)
$H_{(22)c}$	325(1)	741(4)	208(7)
$H_{(23)a}$	219(1)	419(5)	-238(7)
$H_{(23)b}$	206(2)	503(5)	-97(8)
$H_{(23)c}$	223(1)	555(5)	-245(8)

*Hydrogen atoms of the NH_2 group.

2-Amino-4-phenyl-3-cyanopyrido[3′,2′:4,5]thieno[3,2-*b*]pyridines (Ia-Ih). **A.** A mixture of 1 mmole thienopyridine IIIa or IIIb and 0.2 g (3 mmoles) malononitrile in 10 ml pyridine was maintained at 105-110°C for 12 h. After cooling, the mixture was treated with water. The precipitate of Ia or Ib was filtered off, washed with ethanol and hexane, and recrystallized from acetic acid.

B. A sample of 5 ml piperidine was added to a solution of 5 mmoles pyridinethione IIc-IIg in 10 ml ethanol. The mixture was heated to 50°C. Then, 5 mmoles IVa or IVb was added to the solution at this temperature and stirred for 2 min. The precipitate of Ic-Ih wa filtered off, washed with ethanol and hexane, and dried in the air.

Product Ic. ^{13}C NMR spectrum: 13.01 (7-CH_3), 23.98 (9-CH_3), 88.23 ($C_{(3)}$), 116.23, 118.69, 122.54 ($C_{(8)}$), 123.01 (CN, $C_{(4a)}$, $C_{(9a)}$), 123.75 ($C_{Ar\text{-}p}$), 130.44 ($C_{Ar\text{-}o}$), 132.16 ($C_{Ar\text{-}m}$), 134.48 ($C_{Ar\text{-}ipso}$), 147.19, 147.70 ($C_{(2)}$ and $C_{(9b)}$), 153.19 ($C_{(9)}$), 158.63 ($C_{(5a)}$ and $C_{(7)}$), 162.89 ($C_{(4)}$).

2-(2-Aryl-3,3-dicyano-2-propenylthio)-4,6-dimethyl-3-cyanopyridines (Vc and Vh). A sample of 5.1 mmoles IVa or IVb was added to a solution of 5 mmoles pyridinethione IIc in 10 ml ethanol and stirred rapidly for 2 min. The precipitate of Vc or Vh was filtered off, washed with ethanol and hexane, and dried in the air.

Product Vc was obtained in 57% yield, mp 137°C. IR spectrum: 2240 (3-CN), 2224 (CN, side-chain), 1627, 1582, 1548, 1488. ^{1}H NMR spectrum: 2.37 (3H, s, 4CH_3); 2.57 (3H, s, 6-CH_3); 4.90 (2H, s, CH_2); 7.15 (1H, s, 5-H); 7.46-7.80 (4H, m, H_{Ar}). ^{13}C NMR spectrum: 19.60 (4-CH_3), 23.94 (6-CH_3), 34.87 (CH_2), 86.17 ($C(CN)_2$), 104.80 ($C_{(3)}$), 112.64 and 112.88 (side-chain CN), 114.58 (3-CN), 121.31 ($C_{(5)}$), 125.73 ($C_{Ar\text{-}p}$), 130.21 ($C_{Ar\text{-}m}$), 131.39 ($C_{Ar\text{-}o}$), 132.78 ($C_{Ar\text{-}ipso}$), 152.88 ($C_{(4)}$), 158.39 ($C_{(2)}$), 161.83 ($C_{(6)}$), 174.61 ($C{=}C(CN)_2$). Found: C, 55.69; H, 3.31; N, 13.67%. Calculated for $C_{19}H_{13}BrN_4S$: C, 55.76; H, 3.20; N, 13.69%.

Product Vh was obtained in 64% yield, mp 139°C. IR spectrum: 2234 (3-CN), 2220 (side-chain CN), 1593, 1445 cm^{-1}. ^{1}H NMR spectrum: 2.35 (3H, s, 4-CH_3), 2.54 (3H, s, 6-CH_3), 4.90 (2H, s, CH_2), 7.14 (1H, s, 5-H), 7.48-7.60 (5H, m, H_{Ph}), ^{13}C NMR spectrum: 19.60 (4-CH_3), 23.88 (6-CH_3), 34.96 (CH_2), 86.60 ($C(CN)_2$), 104.80 ($C_{(3)}$), 112.86 and 113.10 (side-chain CN), 114.61 (3-CN), 121.27 ($C_{(5)}$), 128.16 ($C_{Ph\text{-}m}$), 132.02 ($C_{Ph\text{-}p}$), 133.63 ($C_{Ph\text{-}ipso}$), 152.86 ($C_{(4)}$), 158.54 ($C_{(2)}$), 161.83 ($C_{(6)}$), 175.79 ($C{=}C(CN)_2$). Found: C, 69.11; H, 4.15; N, 16.91%. Calculated for $C_{19}H_{14}N_4S$: C, 69.07; H, 4.27; N, 16.96%.

5-(4-Bromophenyl)-8,10-dimethylpyrido[3″,2″:4′,5′]thieno[2′,3′:5,6]pyrido[2,3-*d*]pyrimid-4-one (VII). A sample of 5 mmoles Ic in 10 ml formamide was maintained for 2 h at ~100°C. After cooling, the precipitate of VII was filtered off, washed with ethanol and hexane, and dried in the air. The yield was 62°C, mp >300°C. IR spectrum: 3478 (NH), 1680 (CO), 1625, 1586, 1548, 1488, 1438 cm^{-1}. ^{1}H NMR spectrum: 2.60 (3H, s, 8-CH_3), 3.07 (3H, s, 10-CH_3), 7.35 (1H, s, 9-H), 7.67 and 7.93 (4H, d.d, C_{Ar}), 8.61 (1H, s, 2-H). Found: C, 57.14; H, 3.05; N, 13.22%. Calculated for $C_{20}H_{13}N_4OS$: C, 57.02; H, 3.11; N, 13.30%.

This investigation was supported by the Russian Fund for Basic Research (Grant No. 94-03-08823a).

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