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NEW METHOD FOR THE SYNTHESIS OF 6-AMINO-4-ARYL-3,5-DICYANO-3,4-DIHYDROPYRIDINE-2(1H)-THIONES BY RECYCLIZATION OF 4-ARYL-2,6-DIAMINO-4H-THIOPYRANES

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*Ammonium 6-amino-4-aryl-3,5-dicyano-1,4-dihydropyridine-2-thiolates were synthesized by recyclization of 4-aryl-2,6-diamino-3,5-dicyano-4H-thiopyranes in the presence of organic bases. On acidification of the products, the corresponding substituted 3,4-dihydropyridine-2(1H)-thiones were obtained which were used in the synthesis of 2-alkylthio-1,4-dihydropyridines, bis-(pyridyl-2)disulfides and thieno[2,3-*b*]pyridines.*

We previously reported the synthesis of 6-amino-4-aryl-3,5-dicyano-3,4-dihydropyridine-2(1H)-thiones by the interaction of arylmethylenecyanothioacetamides with cyanothioacetamides [1]. In a continuation of this study we have developed a new method for synthesis of these compounds based on recyclization of 4-aryl-2,6-diamino-3,5-dicyano-4H-thiopyranes (Ia-e).

We have shown previously [2, 3] that when the thiopyranes Ia-e are heated they undergo cyclo-elimination to give arylmethylenecyanothioacetamides (IIa-e) and malononitrile, which subsequently react to produce the Michael adducts (IIIa-e). Further intramolecular cyclization of adducts IIIa-e under the influence of base gave substituted 1,4-dihydropyridine-2-thiolates (IVa-e). Treatment of the thiolates IVa-d with 10% aqueous hydrochloric acid gave 6-amino-4-aryl-3,5-dicyano-3,4-dihydropyridine-2(1H)-thiones (Va-d).

Chemical reactions and spectroscopic characteristics confirmed the structures of compounds IVa-e and Va-d. Thus, the thiones Va and Vc reacted with alkyl halides (VIa-e) in basic media to give the sulfides (VIId-h) (method A), which were analogously prepared from the salts IVa,c and e and the halides VIa-h (method B). Compounds VIIh-j underwent the Thorpe-Ziegler reaction to give thieno[2,3-*b*]pyridines (VIIIh-j) (method A), which were also synthesized in one stage from thione Vc and alkyl halides VIe and i (method B). It is interesting that cyclization of the sulfides VII to the derivatives VIII under the influence of aqueous KOH is accompanied by their dehydrogenation.

TABLE 1. Elemental Analysis Results for 4-Aryl-2,6-diamino-3,5-dicyano-4H-thiopyranes (Ia-e)

Compound	Molecular formula	(Found, %) (Calculated, %) C	H	N	S	M.p., °C	Yield, %
Ia	$C_{14}H_{12}N_4OS$	58,98 59,14	4,31 4,25	19,64 19,70	11,04 11,28	189...190	81
Ib	$C_{17}H_{12}N_4S$	66,87 67,08	4,08 3,97	18,47 18,41	10,47 10,53	159...161	75
Ic	$C_{13}H_9ClN_4S$	53,97 54,07	3,05 3,14	19,33 19,40	11,06 11,10	171...172	67
Id	$C_{13}H_9IN_4S$	41,02 41,07	2,36 2,39	14,77 14,74	8,39 8,43	165...167	70
Ie	$C_{14}H_{12}N_4S$	62,61 62,66	4,45 4,51	20,85 20,88	11,88 11,95	170...171	84

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*Deceased.

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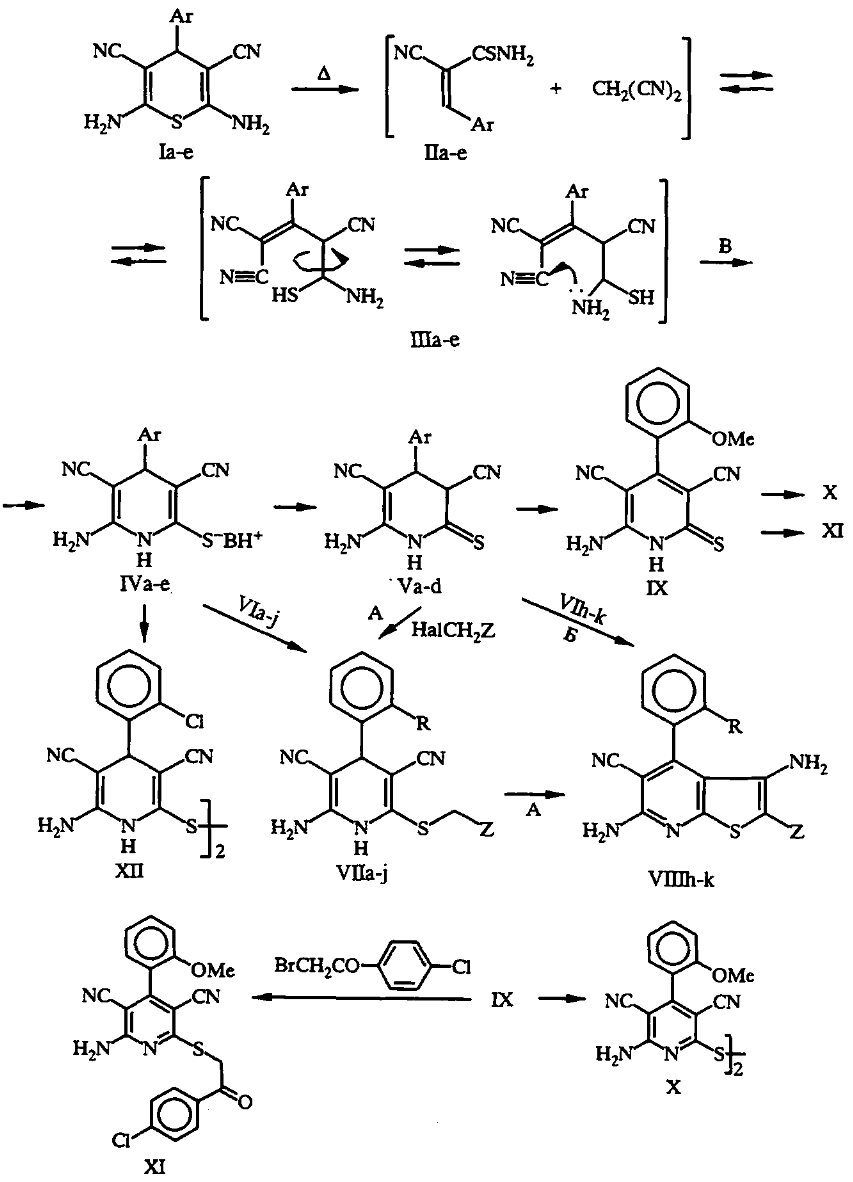
When 6-amino-4-(2-methoxyphenyl)-3,5-dicyano-3,4-dihydropyridine-2(1H)-thione (Va) is boiled in glacial acetic acid, it is converted into the corresponding dehydrogenated analog, the thione IX, which is oxidized by iodine in basic media to the corresponding disulfide X and is alkylated by 4-chlorophenacyl bromide to form the sulfide (XI). The salt IVc reacted with iodine in ethanol to give the corresponding bis[6-amino-3,5-dicyano-4-(2-chlorophenyl)-1,4-dihydro-2-pyridinyl)]disulfide (XII).

The IR spectra of compounds IV, V, VII-XII contain bands characteristic of conjugated cyano groups at 2175-2220 cm^{-1}. Bands corresponding to the amino groups are observed in the 3200-3450 cm^{-1} region (Table 4). Characteristic signals in the ^1H NMR spectra of the substituted dihydropyridines IV, VII, and XII are the broad singlet of the NH proton at 9.01-9.40 ppm, the singlet for the $C_{(4)}$-H proton at 4.60-4.87 ppm, and the broad singlet of the protons of the conjugated amino group at 5.41-5.92 ppm (Table 4), which is particularly indicative of such systems [1].



I—III, V a Ar = 2-$CH_3OC_6H_4$, b Ar = 1-naphthyl, c Ar = 2-ClC_6H_4, d Ar = 2-IC_6H_4,
e Ar = 2-$CH_3C_6H_4$. IV a Ar = 2-$CH_3OC_6H_4$, B = N- methylmorpholine b Ar = 1-naphthyl,
B = morpholine c Ar = 2-ClC_6H_4, B = N- methylmorpholine; d Ar = 2-IC_6H_4, B = N- methylmorpholine
c Ar = 2-$CH_3C_6H_4$, B = N- methylmorpholine
VI a Hal = I, *Z* = H; b Hal = Br, *Z* = CH=CH_2; c Hal = Cl, *Z* = CN; d Hal = Br,
Z = 4-$CH_3C_6H_4CO$; e Hal = Br, *Z* = PhCO; f Hal = Cl, *Z* = 4-BrC_6H_4NHCO; g Hal = Cl,
Z = $COOC_2H_5$; h Hal = Br, *Z* = 4-BrC_6H_4CO; и Hal = Br, *Z* = 2,4-$(CH_3)_2C_6H_3CO$.
VII, VIII a R = CH_3, *Z* = 4-BrC_6H_4NHCO; b R = CH_3, *Z* = H; c R = CH_3, *Z* = $COOC_2H_5$;
d R = CH_3O, *Z* = CH=CH_2; e R = Cl, *Z* = CN; f R = Cl, *Z* = H; g R = Cl, *Z* = 4-$CH_3C_6H_4CO$;
h R = Cl, *Z* = PhCO; i R = CH_3, *Z* = 4-BrC_6H_4CO; j R = CH_3, *Z* = PhCO; k R = Cl,
Z = 2,4-$(CH_3)_2C_6H_3CO$

TABLE 2. IR and ^{1}H NMR Spectra of 4-Aryl-2,6,diamino-3,5-dicyano-4H-thiopyranes (Ia-e)

Compound	IR spectra, ν, cm^{-1}		^{1}H NMR spectra, δ, ppm		
	NH_2	CN	NH_2, s	$C_{(4)}H$, s	Ar, m
Ia	3204, 3320, 3399	2185	6,81	4,58	6,90...7,35, 3,74 s
Ib	3180, 3315, 3422	2172, 2190	6,91	5,21	7,50, 7,91, 8,37
Ic	3300, 3395	2190	6,96	4,82	7,40
Id	3185, 3300, 3422	2175 pl	6,94	4,71	7,36, 7,83 d
Ie	3300, 3420	2200	6,83	4,60	7,16, 2,39 s

EXPERIMENTAL

IR spectra of Nujol mulls were recorded on an IKS-29 spectrometer. ^{1}H NMR spectra of DMSO-D_6 solutions with TMS as internal standard were recorded on a Bruker WP-100 SU (100 MHz) instrument. The course of reactions and the purity of products were monitored by TLC on Silufol UV-254 strips with acetone–hexane (3:5) as eluant.

4-Aryl-2,6-diamino-3,5-dicyano-4H-thiopyranes (Ia-e) were obtained by a known method [4]. Their characteristics are cited in Tables 1 and 2.

N-Methylmorpholinium 6-Amino-4-aryl-3,5-dicyano-1,4-dihydropyridin-2-thiolates (IVa-e). A suspension of thiopyrane Ia-e (10 mmol) in absolute ethanol (20 cm^3) in the presence of the organic base (10 mmol) was boiled for 15 min. The precipitate which formed on cooling the reaction mixture was separated and washed with absolute ethanol and acetone.

N-Methylmorpholinium 6-Amino-4-(2-methoxyphenyl)-3,5-dicyano-1,4-dihydropyridin-2-thiolate (IVa). Yield 77%. M.p. 165-167°C. IR spectrum: 1648 (NH_2), 2170, 2192 (CN), 3210, 3300, 3422 cm^{-1} (NH, NH_2). ^{1}H NMR spectrum: 2.75(3 H, s, NCH_3), 3.14 (4 H, m, CH_2NCH_2), 3.73 (3 H, s, OCH_3), 3.80 (4 H, m, CH_2OCH_2), 4.48 (1 H, s, $C_{(4)}H$), 5.45 (2 H, br. s, NH_2), 6.80-7.20 (4 H, m, Ar), 7.90 ppm (1 H, br. s, NH). Found, %: C 59.11, H 5.88, N 17.99, S 8.45. Calc. for $C_{19}H_{23}N_5O_2S$, %: C 59.20, H 6.01, N 18.17, S 8.32.

Morpholinium 6-Amino-4-(1-naphthyl)-3,5-dicyano-1,4-dihydropyridin-2-thiolate (IVb). Yield 80%. M.p. 188-89°C. IR spectrum: 1600 (NH_2), 2190 (CN), 3225, 3320, 3360 cm^{-1} (NH, NH_2). ^{1}H NMR spectrum: 3.07 (4 H, m, CH_2NCH_2), 3.71 (4 H, m, CH_2OCH_2), 4.82 (1 H, s, $C_{(4)}$), 5.53 (2 H, br. s, NH_2), 7.46-8.50 ppm (8 H, m, naphthyl and NH). Found, %: C 64.60, H 5.50, N 17.76, S 8.02. Calc. for $C_{21}H_{21}N_5OS$, %: C 64.43, H 5.41, N 17.89, S 8.19.

The pyridinethiolates IVc and IVd were identical to those described previously [1]. Yield of IVc 72%, m.p. 183-184°C. For IVd, 80%, 165-166°C.

N-Methylmorpholinium 6-amino-4-(2-methylphenyl)-3,5-dicyano-1,4-dihydropyridin-2-thiolate (IVe). Yield 86%. M.p. 157-60°C. IR spectrum: 1660 (NH_2), 2188 (br., CN), 3305, 3422 cm^{-1} (NH, NH_2). ^{1}H NMR spectrum: 2.31 (3 H, s, CH_3), 2.77 (3 H, s, NCH_3), 3.16 (4 H, m, CH_2NCH_2), 3.76 (4 H, m, CH_2OCH_2), 4.31 (1 H, s, $C_{(4)}H$), 5.47 (2 H, br. s, NH_2), 7.0-7.2 (4 H, m, Ar), 7.92 ppm (1 H, br. s, NH). Found, %: C 61.65, H 6.12, N 19.12, S 8.72. Calc. for $C_{19}H_{23}N_5OS$, %: C 61.76, H 6.27, N 18.95, S 8.68.

6-Amino-4-aryl-,3,5-dicyano-3,4-dihydro-pyridin-2(1H)-thiones (Va-d). A suspension of the salt IVa-d (10 mmol) in ethanol (10 cm^3) was diluted with 10% aqueous hydrochloric acid to pH 4. The solution, which became transparent, was kept for 24 h at 20°C. The precipitate which formed was filtered off and washed with ethanol and hexane.

6-Amino-4-(2-methoxyphenyl)-,3,5-dicyano-3,4-dihydro-pyridin-2(1H)-thione (Va). Yield 63%. M.p. 230-232°C (methanol). IR spectrum: 1715 (NH_2), 2207, 2258 (CN), 3360-3400 cm^{-1} (NH, NH_2). ^{1}H NMR spectrum: 3.65 (3 H, s, CH_3), 4.82 (1 H, d, $C_{(3)}H$, ^3J = 8 Hz), 5.31 (1 H, d, $C_{(4)}H$, ^3J = 8 Hz), 6.9-7.7 ppm (4 H, m, Ar). Found, %: C 58.95, H 4.33, N 19.54, S 11.39. Calc. for $C_{14}H_{12}N_4OS$, %: C 59.14, H 4.25, N 19.70, S 11.28.

6-Amino-4-(1-naphthyl)-,3,5-dicyano-3,4-dihydro-pyridin-2(1H)-thione (Vb). Yield 65%. M.p. 255-257°C (ethanol). IR spectrum: 1710 (NH_2), 2200 (CN), 3215, 3370 cm^{-1} (NH, NH_2). ^{1}H NMR spectrum: 5.43 (1 H, d, $C_{(3)}H$, ^3J = 7 Hz), 5.81 (1 H, d, $C_{(4)}H$, ^3J = 7 Hz), 7.07-8.54 (9 H, m, naphthyl and NH_2), 11.63 ppm (1 H, s, NH). Found, %: C 66.82, H 3.81, N 18.50, S 10.87. Calc. for $C_{17}H_{12}N_4S$, %: C 67.09, H 3.97, N 18.41, S 10.53.

The pyridinethiones Vc and Vd were analogous to those described previously [1]. Yield of Vc 66%, m.p. 270-272°C (ethanol), for Vd 77%, m.p. 278-280°C (ethanol).

TABLE 3. Elemental Analysis Results for Substituted 2-Z-Methylthio-1,4-dihydropyridines (IVa-j), Thieno[2,3-*b*]pyridines (VIIIh-k) and 6-Amino-3,5-dicyano-4(-2-methoxyphenyl)-2-(4-chlorobenzoylmethylthio)pyridine (XI)

Compound	Molecular formula	(Found, %) (Calculated, %) C	H	N	S	M.p., °C (recrystallization solvent)	Yield, %
VIIa	$C_{22}H_{18}BrN_5OS$	55,19 55,01	3,85 3,78	14,41 14,58	6,50 6,67	220...222 (ethanol)	89 (B)
VIIb	$C_{15}H_{14}N_4S$	63,71 63,80	4,85 5,00	19,99 19,84	11,45 11,36	175...177 (ethanol)	70 (B)
VIIc	$C_{18}H_{18}N_4O_2S$	60,95 61,00	5,08 5,12	15,77 15,81	9,01 9,05	145...147 (ethanol)	79 (B)
VIId	$C_{17}H_{16}N_4OS$	62,89 62,94	4,92 4,97	17,24 17,27	9,80 9,88	140...142 (benzene)	66 (A) 72 (B)
VIIe	$C_{15}H_{10}ClN_5S$	55,15 54,96	3,20 3,08	21,21 21,37	9,66 9,78	194...196 (methanol)	80 (A) 82 (B)
VIIf	$C_{14}H_{11}ClN_4S$	55,49 55,54	3,52 3,66	18,70 18,50	10,62 10,59	162...163 (methanol)	77 (A) 79 (B)
VIIg	$C_{22}H_{17}ClN_4OS$	62,81 62,78	3,90 4,07	13,08 13,31	7,80 7,62	193...195 (methanol)	90 (A) 88 (B)
VIIh	$C_{21}H_{15}ClN_4OS$	62,12 61,99	3,81 3,72	13,59 13,77	7,70 7,88	160...162 (methanol)	73 (A) 77 (B)
VII i	$C_{22}H_{17}BrN_4OS$	56,60 56,78	3,59 3,68	12,20 12,04	6,78 6,89	201...203 (ethanol)	78 (B)
VIIj	$C_{22}H_{18}N_4OS$	68,33 68,37	4,65 4,69	14,45 14,50	8,25 8,30	195...197 (ethanol)	77 (B)
VIIIh	$C_{21}H_{13}ClN_4OS$	62,25 62,30	3,20 3,24	13,79 13,84	7,90 7,92	350...352 (AcOH)	75 (A) 64 (B)
VIII i	$C_{22}H_{15}BrN_4OS$	56,84 57,03	3,11 3,26	12,22 12,09	7,08 6,92	300(decomp.) (AcOH)	66 (A)
VIII*l*	$C_{22}H_{16}N_4OS$	68,68 68,73	4,14 4,19	14,53 14,57	8,31 8,34	330(decomp.) (AcOH)	65 (A)
VIIIm	$C_{23}H_{17}ClN_4OS$	63,78 63,81	3,90 3,96	12,91 12,94	7,38 7,41	293...295 (1-butanol)	61 (B)
XI	$C_{22}H_{15}ClN_4O_2S$	60,88 60,76	3,30 3,48	12,95 12,88	7,21 7,37	221...222 (AcOH)	78 (A)

6-Amino-4-aryl-3,5-dicyano-2-Z-methylthio-1,4-dihydropyridines (VIIa-j). A. To a solution of thione Va or Vc (10 mmol) in DMF (10 cm^3) an aqueous 10% KOH solution was added with stirring followed by addition of an alkyl halide (VIa-e, 10 mmol) in 1 min. The reaction mixture was stirred for 2 h and then diluted with water (15 cm^3). The precipitate was filtered off, and washed successively with water, ethanol and hexane to give compounds VIId-h (Tables 3 and 4).

B. An alkyl halide (VIa-h, 10 mmol) was added with stirring to a suspension of salt IVa, c or e (10 mmol) in ethanol (10 cm^3), the mixture was stirred for 1.5 h and then diluted with water (15 cm^3). The precipitate formed was isolated and washed successively with water, ethanol and hexane to give compounds VIIa-j (Tables 3 and 4).

4-Aryl-3,6-diamino-5-cyano-2-Z-thieno[2,2-*b*]pyridines (VIIIh-k). A. A 10% aqueous KOH solution (5.6 cm^3, 10 mmol) was added with stirring to a solution of a substituted 2-alkylthio-1,4-dihydropyridine (VIIh-j, 10 mmol) in DMF (15 cm^3). After stirring for 4 h, the solution was diluted with water (15 cm^3). The precipitate formed was isolated and washed successively with water, ethanol and hexane to give compounds VIIIh-j (Tables 3 and 4)

B. A 10% aqueous KOH solution (5.6 cm^3, 10 mmol) was added with stirring to a solution of thione Vc (2.89 g, 10 mmol) in DMF (15 cm^3). An alkyl halide (VIe or VIi, 10 mmol) was added after 1 min. More 10% aqueous KOH solution (5.6 cm^3) was added over 30 min to the reaction mixture, which was stirred for a further 4 h and then diluted with water (15 cm^3). The precipitate formed was isolated and washed successively with water, ethanol and hexane to give compounds VIIIh and k (Tables 3 and 4).

6-Amino-3,5-dicyano-4-(2-methoxyphenyl)pyridin-2(1H)-thione (IX). A suspension of compound Va (2.84 g, 10 mmol) in glacial acetic acid (10 cm^3) was heated to boiling and filtered hot. The precipitate which formed on cooling the mixture was separated and washed with glacial acetic acid and ether to give the thione IX, 2.03 g (72%). M.p. 238-240°C (AcOH). IR spectrum: 1650 (NH_2), 2220 (CN), 3200, 3304, 3408 cm^{-1} (NH_2). ^{1}H NMR spectrum: 3.81 (3 H, s, CH_3), 7.0-7.6 (4 H, m, Ar), 7.93 ppm (2 H, br.s, NH_2). Found %: C 59.44, H 3.69, N 19.76, S 11.49. Calc. for $C_{14}H_{10}N_4OS$, %: C 59.56, H 3.57, N 19.85, S 11.36.

TABLE 4. IR and ^{1}H NMR Spectra of Substituted 2-Z-Methylthio-1,4-dihydropyridines (VIIa-j), Thieno[2,3-*b*]pyridines (VIIIh-k) and 6-Amino-3,5-dicyano-4(-2-methoxyphenyl)-2-(4-chlorobenzoylmethylthio)pyridine (XI)

Compound	IR spectra, ν, cm^{-1}		^{1}H NMR spectra, δ, ppm					
	NH, NH$_2$	CN	NH$_2$, s	NH, s	Ar, m	C$_{(4)}$H, s	SCH$_2$, s, C$_{(3)}$—NH$_2$, br.s.	Z
VIIa	3210, 3300, 2420	2190, 2218	5,92	9,40	7,14 m, 2,34 s	4,61	3,91	10,43 s, 7,54 m
VII b	3150...3415	2175, 2220	5,91	9,01	7,17 m, 2,35 s	4,61	2,50	—
VIIc	3195, 3308, 3444	2220	5,85	9,25	7,17 m, 2,36 s	4,63	3,87	4,11 q 1,19 t
VIId	3160, 3320, 3435	2190	5,83*	9,13	7,02, 3,74*	4,59	*	*
VIIe	3250, 3372	2170, 2195	8,41	9,95	7,40	4,87	4,22	—
VIIf	3200, 3332, 3395	2190 pl.	6,04	9,12	7,35	4,81	2,51	—
VIIg	3225, 3384, 3442	2192 pl. 2220 pl.	5,97	9,31	7,36	5,01	4,72	7,91 m, 2,40 s
VIIh	3322, 3440	2180 pl.	5,98	9,28	7,38	4,80	4,60	7,60 m, 8,02 m
VIIi	3300...3424	2192 pl.	5,83	9,20	7,17 m, 2,34 s	4,63	4,69	7,45 d, 7,78 d
VIIj	3300...3422	2195 pl.	5,85	9,19	7,62 m, 2,33 s	4,60	4,73	7,16 m
VIIIh	3205, 3304, 3422	2220	7,64*	—	*	—	6,61	*
VIII i	3300, 3400	2218	7,50*	—	*, 2,11	—	6,53	7,65 m
VIIIj	3333, 3412	2200 pl.	7,52*	—	*, 2,13 s	—	6,54	7,70 m
VIIIk	3150, 3268, 3470	2220	7,71*	—	*	—	6,48	7,26 d, 7,10 m, 2,31 s, 2,22 s
XI	3222, 3314, 3398	2220	7,64	—	7,00...7,55 m, 3,81 s	—	4,96	8,08 d, 7,65 d

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*Signals overlapped.

Bis[(6-amino-3,5-dicyano-4-(2-methoxyphenyl)-2-pyridinyl) Disulfide (X). A 10% aqueous KOH solution (1.28 cm^3, 2.28 mmol) was added to a stirred suspension of thione IX (0.64 g, 2.28 mmol) in ethanol (10 cm^3), followed by dropwise addition of iodine (0.58 g, 2.28 mmol) in ethanol (15 cm^3). After 1 h, water was added (10 cm^3) and the precipitate was isolated to give compound X (0.58 g, 91%). M.p. 154-156°C (ethanol). IR spectrum: 1652 (NH$_2$), 2170, 2218 (CN), 3333, 3425 cm^{-1} (NH$_2$). ^{1}H NMR spectrum: 3.86 (6 H, s, 2CH$_3$), 7.0-7.7 (8 H, m, 2Ar), 7.93 ppm (4 H, br.s, NH$_2$). Found, %: C 59.88, H 3.40, N 19.80, S 11.25. Calc. for $C_{28}H_{18}N_8O_2S_2$, %: C 59.77, H 3.23, N 19.92, S 11.40

6-Amino-,3,5-dicyano-4-(2-methoxyphenyl)-2-(4-chlorobenzoylmethylthio)pyridine (XI) was prepared analogously to that for compound VII by method A. Its characteristics are cited in Tables 3 and 4.

Bis[(6-amino-3,5-dicyano-4-(2-chlorophenyl)-1,4-dihydro-2-pyridyl)] Disulfide (XII). A solution of iodine (0.56 g, 2.28 mmol) in ethanol (15 cm^3) was added dropwise with stirring to a suspension of salt IVc (0.89 g, 2.28 mmol) in ethanol (10 cm^3). After 1 h, the mixture was diluted with water (10 cm^3) and the precipitate isolated to give disulfide XII (0.49 g, 74%). M.p. 165-166°C (ethanol). IR spectrum: 1635 (NH$_2$), 2180 (br, CN), 3200, 3330, 3414 cm^{-1} (NH$_2$). ^{1}H NMR spectrum: 4.91 (2 H, br.s, $2C_{(4)}H$), 5.99 (4 H, br.s, $2NH_2$), 7.45 (8 H, m, 2Ar), 9.67 ppm (2 H, br.s, 2NH). Found %, C 54.12, H 2.88, Cl 12.15, N 19.65, S 11.20. Calc. for $C_{26}H_{16}Cl_2N_8S_2$, %: C 54.26, H 2.80, Cl 12.32, N 19.47, S 11.14.

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