Diversity-oriented Cascade Synthesis of Pyrido[2',3':4,5]thieno[2,3-b]pyridine Derivatives

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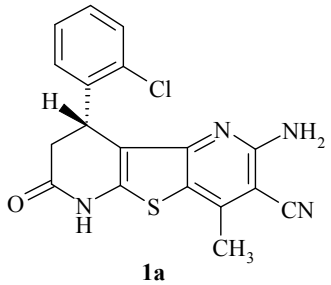
Abstract: A 20-membered library of 2-amino-9-aryl-3-cyano-4-methyl-7-oxo-6,7,8,9-tetrahydropyrido[2',3':4,5]thieno[2,3-b]pyridines which recently have been reported as selective progesterone receptor agonists was synthesized by ternary condensation of N-methylmorpholinium 4-aryl-3-cyano-6-oxo-1,4,5,6-tetrahydropyridine-2-thiolates with malononitrile and acetone, and alternatively – by reaction of *bis*(4-aryl-3-cyano-6-oxo-1,4,5,6-tetrahydropyridine-2-yl)disulfides with acetone and malononitrile in basic media. The mechanism, scope and limitations of the reactions are discussed.

Keywords: dipyridothiophenes, thienopyridines, progesterone receptor agonists, cascade heterocyclization.

Introduction

Thieno[2,3-b]pyridine derivatives are of significant importance due to their practical usefulness (for reviews, please see [1-3]). Although the synthesis and properties of thieno[2,3-b]pyridines are well documented, their condensed analogs were, in our opinion, studied very little. As we have reported previously [4-9], ternary condensation of acetone and malononitrile with a variety of pyridine-2-thiolates and pyridine-2(1*H*)-thiones leads to 2-amino-9-aryl-3-cyano-4-methylpyrido[2',3' : 4,5]thieno[2,3-b]pyridines **1-5** (Scheme 1). Recently, dipyridothiophenes **1** and, in particular, (*S*)-2-amino-9-(2-chlorophenyl)-3-cyano-4-methyl-7-oxo-6,7,8,9-tetrahydropyrido[2',3' : 4,5]thieno[2,3-b]pyridine (**1a**, Fig. 1) have been reported as powerful and selective non-steroidal progesterone receptor agonists [10].

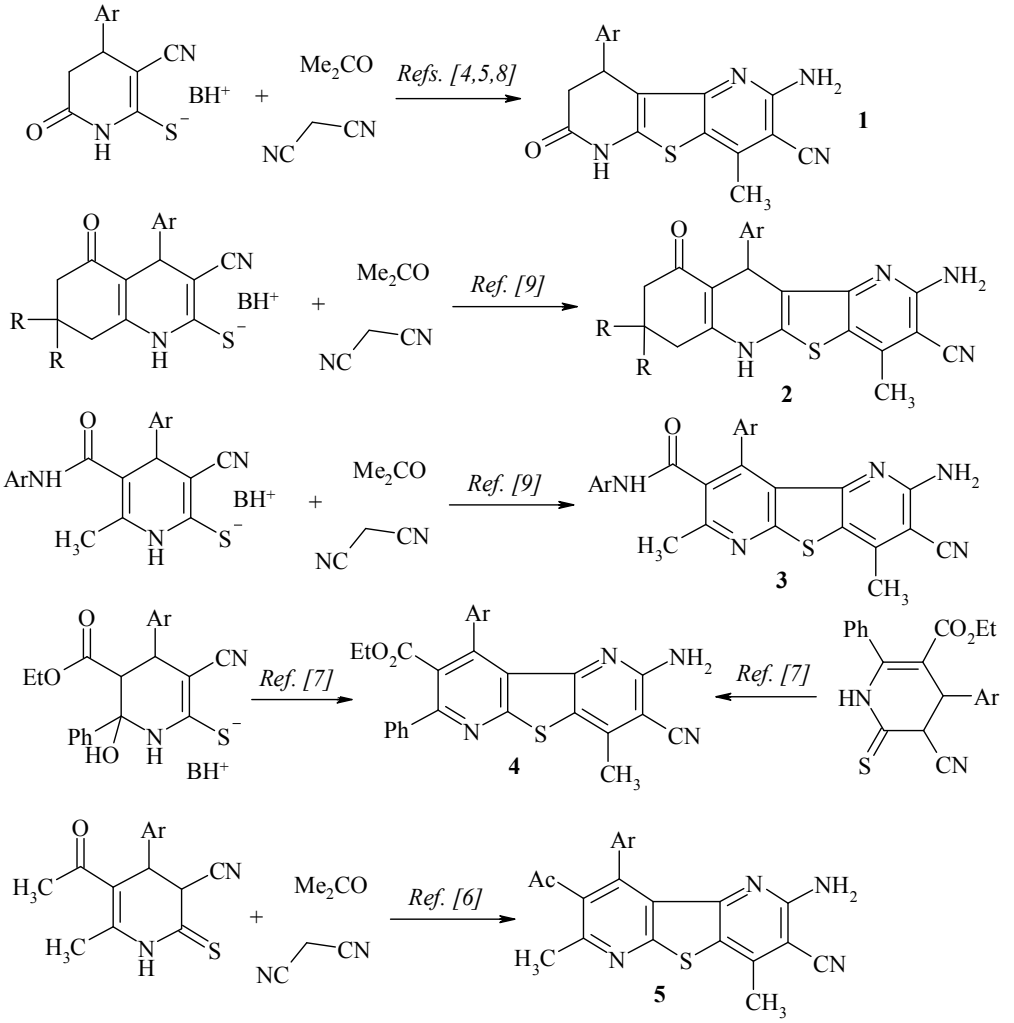
Fig. 1. (*S*)-2-Amino-9-(2-chlorophenyl)-3-cyano-4-methyl-7-oxo-6,7,8,9-tetrahydropyrido[2',3' : 4,5]thieno[2,3-b]pyridine



1a

Herein, we report the synthesis of novel tetrahydropyrido[2',3':4,5]-thieno[2,3-b]pyridine analogs of (**1a**), synthesized from thiolates **6**, or, alternatively, by ternary condensation of bis(2-pyridyl)disulfides **7** with malononitrile and acetone.

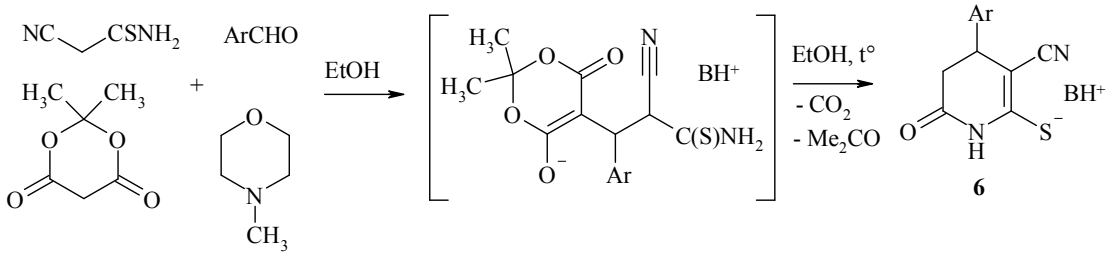
Scheme 1:



B = *N*-methylmorpholine.

Results and Discussion

Starting *N*-methylmorpholinium 4-aryl-3-cyano-6-oxo-1,4,5,6-tetrahydropyridine-2-thiolates **6** were obtained as mixtures of (4*S*)- and (4*R*)-isomers in 1:1 ratio by one-pot condensation of cyanothioacetamide, aromatic aldehyde and Meldrum's acid in the presence of excessive N-methylmorpholine [5,11,12] as follows (Scheme 2):

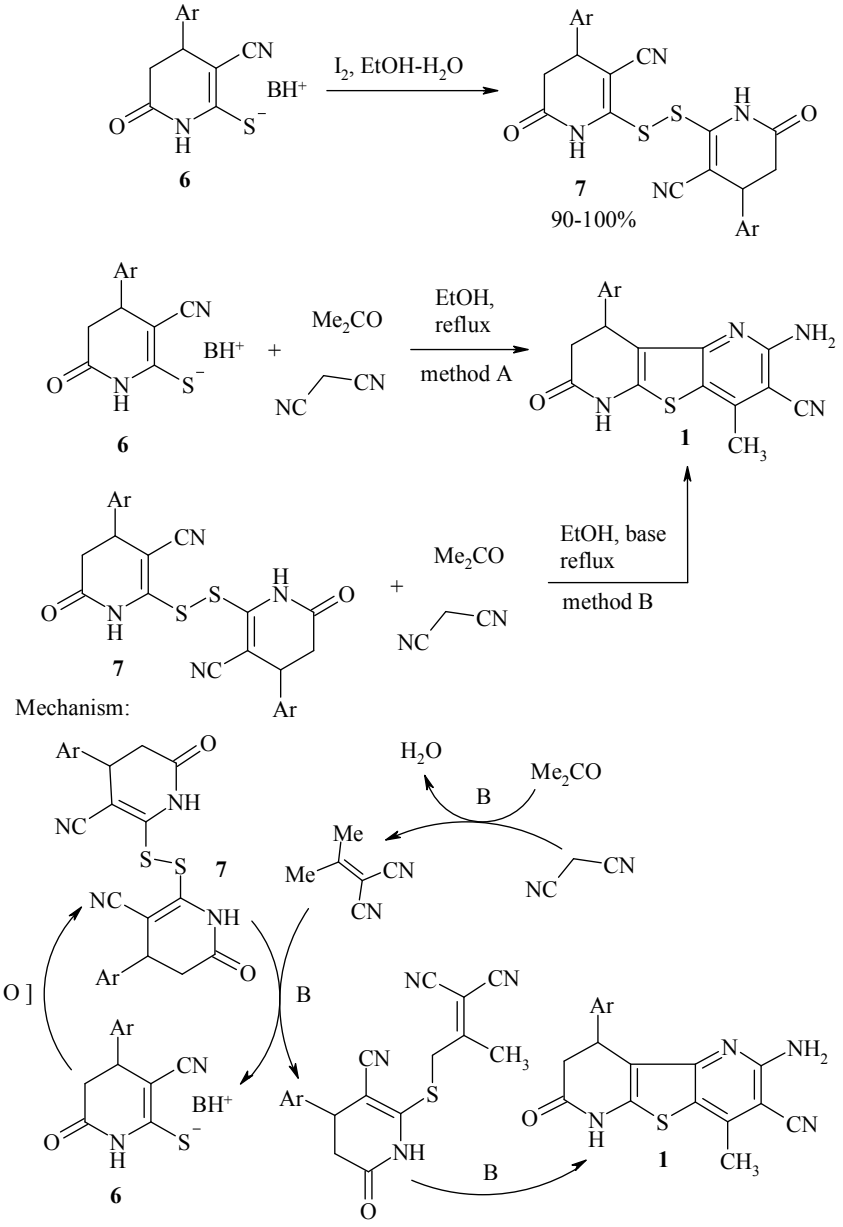


B = *N*-methylmorpholine.

We have examined the reactions of thiolates **6** with malononitrile and acetone. When treated with malononitrile and excessive acetone (*cca*. 10 eq.) in refluxing EtOH, thiolates **6** reacted to form desired dipyridothiophenes **1** in low to moderate yields (method A, Scheme 3). As we have shown earlier [8], the mechanism of this unusual reaction involves the oxidation of the starting thiolates

by atmospheric oxygen to give bis(2-pyridyl)disulfides **7**, subsequent nucleophilic cleavage of the S–S bond by the isopropylidenemalononitrile anion and final cascade heterocyclization (Scheme 3). So we attempted to synthesize compounds **1** by reaction of *bis*(4-aryl-3-cyano-6-oxo-1,4,5,6-tetrahydropyridine-2-yl)disulfides **7** with acetone and malononitrile in the presence of tertiary base (Et_3N, N-methylmorpholine) (Method B, Scheme 3). Disulfides **7** were prepared quantitatively by mild oxidation of thiolates **6** with iodine in aqueous EtOH [8]:

Scheme 3.

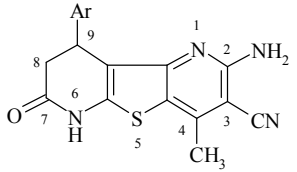


B = *N*-methylmorpholine.

The yields and ^{1}H NMR spectra data are given in Table 1.

Method B has been found to be superior to method A in terms of efficiency, higher yields and decrease of reaction time. 2-Amino-9-aryl-3-cyano-4-methyl-7-oxo-6,7,8,9-tetrahydropyrido[2',3':4,5]thieno[2,3-b]pyridines are stable crystalline solids with colours range from almost white to yellow, insoluble in EtOH, THF or ether, sparingly soluble in acetone, and readily soluble in DMSO, DMF or hot AcOH.

Table 1. Properties and ^{1}H NMR data (200 MHz) for compounds **1**.



1

Compound, appearance	Ar	Yields, % (method)	M.p. (solvent)	^{1}H NMR data (δ, *J*, Hz)					
				Ar	C(8)H$_2$**	C(9)H*	CH_3	NH_2	NH
1a pale-yellow crystals	2-ClC_6H_4	37 (A) [4] 62 (B)	297-298 (AcOH)	6.67, 7.11-7.28, 7.47 (three m, 4 H)	2.70, 3.13 (both m, each 1 H)	4.84 (m, 1 H)	2.54 (s, 3 H)	6.28 (br.s, 2 H)	11.15 (s, 1 H)
1b yellow crystals	2,3-$(MeO)_2C_6H_4$	36 (A) [4] 77 (B)	> 300 (AcOH)	3.80,3.89 (both s, each 3 H, 2 MeO); 6.27, 6.79 (both m, 3 H)	2.97-3.10 (m, 2 H)	4.73 (br. pseudo-d, 1 H)	2.47 (s, 3 H)	6.08 (br.s, 2 H)	10.96 (s, 1 H)
1c white powder	2-$MeOC_6H_4$	29 (A) [5] 77 (B)	> 300 (AcOH)	3.91 (s, 3 H, MeO); 6.54 (d, ^3J = 7.5), 6.72 (m), 6.96 (d, ^3J = 8.1), 7.18 (m, 4 H)	2.67 (br. pseudo-d, 1 H, ^2J = 16.6); 3.02 (dd, 1 H, ^2J = 16.6, ^3J = 8.0)	4.72 (br. pseudo-d, 1 H)	2.52 (s, 3 H)	6.23 (br.s, 2 H)	10.97 (s, 1 H)
1d yellow crystals	1-naphthyl	36 (A) [5] 73 (B)	> 300 (dec., AcOH)	6.73-8.31 (m, 7 H)	2.72 (br.pseudo-d, 1 H, ^2J = 16.0); 3.32 (m, 1 H)	5.34 (br. pseudo-d, 1 H)	2.54 (s, 3 H)	6.35 (br.s, 2 H)	11.13 (br.s, 1 H)
1e pale-yellow crystals	4-$EtOC_6H_4$	30 (A) [5] 66 (B)	262-263 (EtOH-AcOH, 2 : 1)	1.32 (t, 3 H, EtO, ^3J = 6.9); 3.91 (q, 2 H, EtO, ^3J = 6.9); 6.70, 7.02 (both d, 2 H each, ^3J = 8.3)	2.67, 2.99 (both br. pseudo-d, 1 H each, ^2J = 16.2)	4.46 (br. pseudo-d, 1 H)	2.49 (s, 3 H)	6.18 (br.s, 2 H)	10.97 (br.s, 1 H)
1f pale-yellow crystals	3,4-$(MeO)_2C_6H_4$	38 (A) [5] 79 (B)	298 (EtOH-AcOH, 1 : 1)	3.70 and 3.74 (both s, 3 H each, $(MeO)_2$) 6.55 and 6.70 (both d, 1 H each, ^3J = 8.2); 7.02 (s, 1 H)	2.76 and 3.02 (both br. pseudo-d, 1 H each, ^2J = 16.6)	4.48 (br. pseudo-d, 1 H)	2.51 (s, 3 H)	6.19 (br.s, 2 H)	10.99 (br.s, 1 H)
1g pale-yellow	2,5-$(MeO)_2C_6H_4$	35 (A) [5]	> 300 (EtOH-	3.59 and 3.86 (both s, 3 H each,	2.66 (br. pseudo-d, 1 H,	4.68 (br. pseudo-d, 1 H)	2.53 (s, 3 H)	6.04 (br.s, 2 H)	10.93 (br.s, 1 H)

crystals			AcOH, 1 : 2)	$(MeO)_2$); 6.12 (s, 1 H) 6.64 and 6.82 (both d, 1 H each, Ar, ^3J = 8.6)	^2J = 16.6); 2.93 (dd, 1 H, ^2J = 16.6, ^3J = 7.8)				
1h pale-yellow crystals	4-MeC_6H_4	34 (A) [5] 77 (B)	288-290 (EtOH-AcOH, 1 : 3)	2.19 (s, 3 H, Me); 6.96 (br.s, 4 H)	2.68 and 3.00 (both br. pseudo-d, 1 H each, ^2J = 16.0)	4.41 (br. pseudo-d, 1 H)	2.43 (s, 3 H)	6.06 (br.s, 2 H)	10.91 (br.s, 1 H)
1i finely crystalline white powder	3,4,5-$(MeO)_3$-C_6H_2	28 (A) [5]	276-278 (AcOH)	3.59 (s, 3 H, MeO); 3.69 (s, 6 H, $(MeO)_2$); 6.56 (s, 2 H)	2.78 and 3.02 (both br. pseudo-d, 1 H each, ^2J = 16.3)	4.45 (br. pseudo-d, 1 H)	2.47 (s, 3 H)	6.28 (br.s, 2 H)	11.01 (br.s, 1 H)
1j pale-yellow crystals	2-thienyl	27 (A) [5] 70 (B)	297-299 (AcOH), solvate with AcOH (1 : 1)	6.72, 6.82, 7.12 (three m, 3 H, thienyl)	2.80 (br. pseudo-d, 1 H, ^2J = 16.3), 3.08 (dd, 1 H, ^2J = 16.3, ^3J = 7.1)	4.70 (br. pseudo-d, 1 H)	1.89 (s, 3 H, CH_3COOH); 2.48 (s, 3 H)	6.24 (br.s, 2 H)	11.03 (br.s, 1 H)
1k colorless crystals	3,4-(OCH_2O)-C_6H_3	30 (A) [5] 64 (B)	276-278 (AcOH-EtOH, 4 : 1)	5.10 (br.s, 2 H, OCH_2O); 6.53-6.73 (m, 3 H)	2.67 and 2.98 (both br. pseudo-d, 1 H each, ^2J =16.2)	4.44 (br. pseudo-d, 1 H)	2.50 (s, 3 H)	6.25 (br.s, 2 H)	11.01 (br.s, 1 H)
1l colorless crystals.	C_6H_5	51 (A) [5] 74 (B)	> 300 (EtOH-AcOH, 1 : 1)	7.12-7.26 (m, 5 H, Ph)	2.70 (br. pseudo-d, 1 H, ^2J = 16.2), 3.08 (m, 1 H)	4.52 (br. pseudo-d, 1 H)	2.49 (s, 3 H)	6.28 (br.s, 2 H)	11.01 (br.s, 1 H)
1m yellow crystals	4-$NO_2C_6H_4$	41 (A) 63 (B)	> 300 (AcOH)	7.43, 8.07 (both d, 2 H each, ^3J = 8.7)	2.73 (br. pseudo-d, 1 H, ^2J = 16.3), 3.16 (m, 1 H)	4.64 (br. pseudo-d, 1 H)	2.50 (s, 3 H)	6.26 (br.s, 2 H)	11.13 (br.s, 1 H)
1n yellowish powder	2-MeC_6H_4	42 (A)	> 250 (AcOH)	2.48 (s, 3 H, Me); 6.60 (d, 1 H, ^3J = 7.5); 6.95-7.22 (m, 3 H)	2.37 (br. pseudo-d, 1 H, ^2J = 13.7), 3.20 (dd, 1 H, ^2J = 13.7, ^3J = 8.6)	4.63 (br. pseudo-d, 1 H)	2.49 (s, 3 H)	6.44 (br.s, 2 H)	11.15 (s, 1 H)
1o pale-yellow crystals	4-$PhCH_2O$-C_6H_4	46 (A)	276-278 (AcOH)	4.99 (s, 2 H, $OC\underline{H}_2Ph$); 6.81, 7.04 (both d, 2 H each, ^3J = 8.4, C_6H_4); 7.24-7.37 (m, 5 H, Ph)	2.68 (br. pseudo-d, 1 H, ^2J = 15.9), 3.00 (m, 1 H)	4.46 (br. pseudo-d, 1 H)	2.49 (s, 3 H)	6.23 (br.s, 2 H)	10.99 (s, 1 H)

1p pale-yellow crystals	3-$MeOC_6H_4$	44 (A)	306-308 (AcOH-EtOH, 3 : 1)	3.70 (s, 3 H, MeO); 6.66-6.79 (m, 3 H); 7.12 (dd, 1 H, ^3J = 7.8, ^3J = 8.0)	2.73 (br. pseudo-d, 1 H, ^2J = 16.1), 3.07 (m, 1 H)	4.49 (br. pseudo-d, 1 H)	2.49 (s, 3 H)	6.23 (br.s, 2 H)	11.01 (s, 1 H)
1q pale-yellow crystals	4-ClC_6H_4	38 (A) 77 (B)	328-329 (AcOH)	7.13, 7.32 (both d, 2 H each, ^3J = 8.4)	2.66 (br. pseudo-d, 1 H, ^2J = 16.6), 3.20 (dd, 1 H, ^2J = 16.6, ^3J = 8.5)	4.48 (br. pseudo-d, 1 H)	2.45 (s, 3 H)	6.58 (br.s, 2 H)	11.16 (s, 1 H)
1r beige powder	4-FC_6H_4	38 (A) 69 (B)	308 (AcOH)	7.04, 7.20 (both m, 2 H each)	2.71 (br. pseudo-d, 1 H, ^2J = 16.1), 3.10 (m, 1 H)	4.53 (br. pseudo-d, 1 H)	2.51 (s, 3 H)	6.27 (br.s, 2 H)	11.04 (s, 1 H)
1s pale-yellow crystals	4-HO-3-MeO-C_6H_3	34 (A)	> 300 (EtOH-AcOH, 1 : 1)	3.72 (s, 3 H, MeO); 6.38 (dd, 1 H, ^4J = 1.8, ^3J = 8.2, H(6) aryl); 6.57 (d, 1 H, ^3J = 8.2, H(5) aryl); 6.91 (d, 1 H, ^4J = 1.8); 8.27 (br.s, 1 H, OH)	2.70 (br. pseudo-d, 1 H, ^2J = 16.5), 3.02 (dd, 1 H, ^2J = 16.5, ^3J = 7.6)	4.42 (br. pseudo-d, 1 H)	2.48 (s, 3 H)	6.29 (br.s, 2 H)	10.96 (s, 1 H)
1t pale-yellow crystals	2-FC_6H_4	30 (A) 71 (B)	268-270 (EtOH-AcOH, 1 : 1)	6.66 (m, 1 H); 6.91-7.22 (m, 3 H)	2.59 (br. pseudo-d, 1 H, ^2J = 16.6), 3.09 (dd, 1 H, ^2J = 16.6, ^3J = 8.3)	4.71 (br. pseudo-d, 1 H)	2.48 (s, 3 H)	6.17 (br.s, 2 H)	11.06 (s, 1 H)

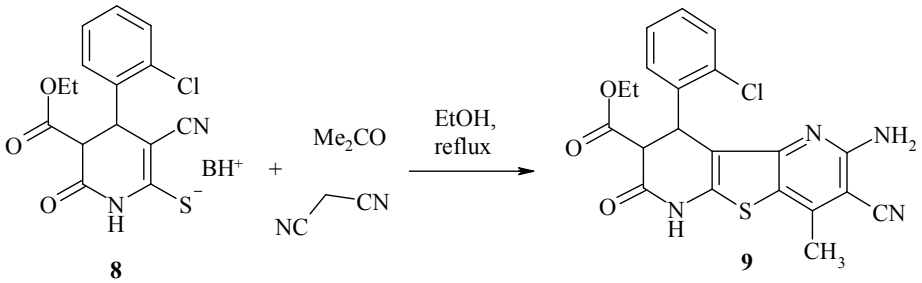
* Signals of C(9)H protons were not resolved as *dd* but appeared as broadened pseudo-doublets.

** Signals of $C(8)H_2$ protons should appear as pair of *dd* with coupling constants ^2J = 15-16 Hz, ^3J = 7-9 Hz for *trans*-H and ^3J = 2-4 Hz for *cis*-H. Actually they appear as pseudo-doublet (unresolved dd) for *cis*-C(8)H and as multiplet or *dd* for *trans*-C(8)H.

The structures of the compounds obtained were confirmed by IR, ^{1}H-spectroscopy as well as elemental analysis data. The IR spectra revealed C≡N stretching at 2205-2190 cm^{-1}, characteristic stretching bands of the C(2)NH$_2$ group and the endocyclic N(6)H group (3550-3145 cm^{-1}) and absorption bands of C=O group in the region of 1692-1670 cm^{-1}.

We also attempetd to broaden the scope of the reaction to include other pyridine-2-thiolates such as **8** [13]. It was found that the latter reacted with acetone and malononitrile under similar conditions to afford corresponding dipyridothiophene **9**, albeit in low yield (13%) (Scheme 4):

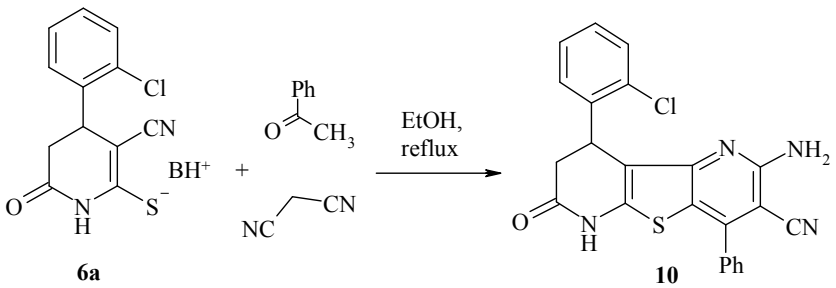
Scheme 4.



8 **9**

Next, we proceeded to explore the reagent scope of this new reaction. However, we failed to obtain any dipyridothiophene species by replacement of acetone and malononitrile with other ketones and active methylene nitriles. Only 4-phenyl derivative **10** was prepared in 27% yield by condensation of thiolate **6a** with acetophenone and malononitrile (Scheme 5):

Scheme 5.



6a **10**

Conclusions

In summary, we synthesized a small library of 2-amino-9-aryl-3-cyano-4-methyl-7-oxo-6,7,8,9-tetrahydropyrido[2',3':4,5]thieno[2,3-b]pyridines by ternary condensation of N-methylmorpholinium 4-aryl-3-cyano-6-oxo-1,4,5,6-tetrahydropyridine-2-thiolates with malononitrile and acetone (method A), and by reaction of *bis*(4-aryl-3-cyano-6-oxo-1,4,5,6-tetrahydropyridine-2-yl)disulfides with acetone and malononitrile in basic media (method B). The method B found to be preferable.

Experimental

Melting points were measured on a Koefler hot stage. Elemental analyses for C, H, and N were conducted using Perkin-Elmer CHN analyzer. IR spectra were recorded on an IKS-29 spectrophotometer in Nujol mulls. The ^{1}H NMR spectra were performed on Varian Gemini 200 (199.975 MHz) spectrometer in DMSO-d$_6$

solutions. The purity of all obtained compounds was checked by TLC on Silufol® UV 254 plates (sorbent – Silpearl, large-pore silicagel after Pitra with luminiscent indicator for UV 254 on the aluminium foil, binder – starch) in the acetone–hexane (1:1) system; spots were visualized with iodine vapors and UV light.

***N*-Methylmorpholinium 4-aryl-3-cyano-6-oxo-1,4,5,6-tetrahydropyridine-2-thiolates (6)** were obtained by improved procedure [5,11,12] as follows: to a mixture of 10.0 g (0.1 mol) of cyanothioacetamide and 0.1 mol of corresponding aldehyde in 40 ml of EtOH ten drops of N-methylmorpholine were added. The reaction mixture was stirred for 1 h, then 50 ml of EtOH, 14.4 g (0.1 mol) of Meldrum's acid and 16.5 ml (0.15 mol) of N-methylmorpholine were added in sequence at vigorous stirring. The reaction appeared to be complete in about 5-10 min to give the Michael adduct as white precipitate. The suspension of the adduct was refluxed for 3-4 h with stirring (in the beginning, CO_2 evolved extensively) to form orange-red solution, which was evaporated to ½. The residue solidified when treated with 50 ml of acetone and stirred for another 3-4 h at room temperature. Yellow or slightly orange precipitate was filtered off, washed with EtOH, acetone and ether to give pure thiolates **6** in 55-80% yields.

Synthesis of *bis*(4-aryl-3-cyano-6-oxo-1,4,5,6-tetrahydropyridine-2-yl)disulfides (7). General procedure. To the stirred suspension of pyridine-2-thiolate **6** (10 mmol) in aq. 90% EtOH (30 ml), iodine (1.28 g, 5 mmol) was added. The mixture was heated to the boiling point and then left to stand at room temperature for 24 h. Water (15 ml) was added dropwise to the reaction mixture; the precipitate was filtered off, washed with aqueous EtOH and dried at 60 °C. Resulting crude disulfides **7** were put into the reaction without any further purification.

2-Amino-9-aryl-3-cyano-7-oxo-6,7,8,9-tetrahydropyrido[2',3':4,5]thieno-[2,3-b]pyridines (1, 9, 10). General procedures.

Method A. A suspension of corresponding thiolate **6** (8 mmol), malononitrile (0.81 g, 12.3 mmol), and excessive acetone (6 ml, 82 mmol) in 35 ml of EtOH was refluxed for 10-20 h. The reaction mixture was kept at 20 °C for 24 h, and the crystalline product was filtered off, washed with a plenty of EtOH, and recrystallized from an appropriate solvent (see Table 1).

Method B. A mixture of disulfide **7** (4 mmol), malononitrile (0.79 g, 12 mmol), acetone (5.9 ml, 80 mmol) and an organic base (N-methylmorpholine or Et_3N) (12 mmol) in 96% EtOH (25 ml) was heated under reflux for 8 h and then kept at room temperature for 24 h. The resulting precipitate of dipyridothiophene **1** was filtered off, washed with EtOH and recrystallised from an appropriate solvent.

2-Amino-9-(2-chlorophenyl)-3-cyano-8-ethoxycarbonyl-4-methyl-7-oxo-6,7,8,9-tetrahydropyrido[2',3':4,5]thieno[2,3-b]pyridine (9). Yield 13% (method A), mp > 250 ºC. IR, ν, cm^{-1}: 3570-3270 (NH, NH_2), 2190 (C≡N), 1720, 1680 (2 C=O). ^{1}H NMR (200 MHz), δ, ppm (*J*, Hz): 1.21 (t, 3 H, ^3J = 7.0, $C\underline{H}_3CH_2$); 2.55 (s, 3 H, C(4)Me); 3.64 (br.s (unresolved d), 1 H, C(9)H); 4.16 (q, 2 H, ^3J = 7.0, $CH_3C\underline{H}_2$); 5.19 (br.s (unresolved d), 1 H, C(8)H); 6.21 (br.s, 2 H, NH_2); 6.67 (m, 1 H, Ar); 7.13-7.27 (m, 2 H, Ar); 7.48 (m, 1 H, Ar); 11.43 (s, 1 H,

NH). Found, %: C 57.84; H 3.92; N 12.90. $C_{21}H_{17}ClN_4O_3S$ (M = 440.91). Calculated, %: C 57.21; H 3.89; N 12.71.

2-Amino-9-(2-chlorophenyl)-3-cyano-4-phenyl-7-oxo-6,7,8,9-tetrahydropyrido[2',3':4,5]thieno[2,3-b]pyridine (10). Yield 27% (method A), decomp. > 310 ºC. IR, ν, cm^{-1}: 3540-3330 (NH, NH_2), 2206 (C≡N), 1678 (C=O). ^{1}H NMR (200 MHz), δ, ppm (*J*, Hz): 2.70 (br. pseudo-d, 1 H, ^2J = 16.0, C(8)H_{cis}), 3.10 (m, 1 H, C(8)H_{trans}); 4.87 (br. pseudo-d, 1 H, C(9)H); 6.28 (br.s, 2 H, NH_2); 6.71 (m, 1 H, Ar); 7.10-7.27 (m, 2 H, Ar); 7.44 (m, 1 H, Ar); 7.59 (m, 5 H, Ph); 11.13 (s, 1 H, NH). Found, %: C 63.84; H 3.56; N 13.14. $C_{23}H_{15}ClN_4OS$ (M = 430.92). Calculated, %: C 64.11; H 3.51; N 13.00.

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