Synthesis of substituted 2-amino-3-cyano-7,9-dimethyl-4*H*-pyrano[2´,3´:4,5]thieno[2,3-*b*]pyridines

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4,6-Dimethyl-2*H*-thieno[2,3-*b*]pyridin-3-one reacts with 2-aryl-1,1-dicyanoethylenes or an aromatic aldehyde/ketone (cyclohexanone and piperidone derivatives) and malononitrile to give substituted 2-amino-3-cyano-7,9-dimethyl-4*H*-pyrano[2´,3´:4,5]thieno[2,3-*b*]pyridines.

Key words: 4,6-dimethyl-2*H*-thieno[2,3-*b*]pyridin-3-one, 2-aryl-1,1-dicyanoethylenes, malononitrile, three-component condensation.

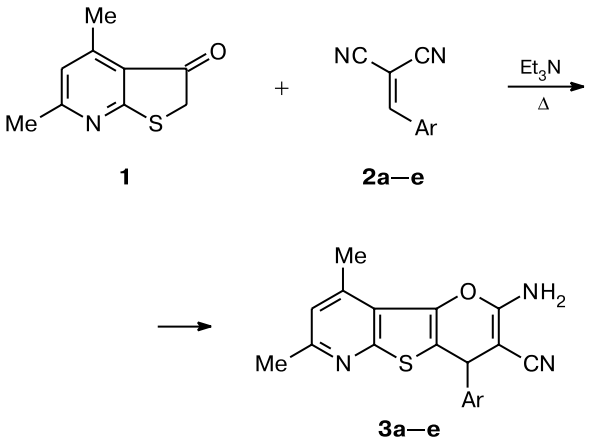
Regio- and stereoselective methods for the synthesis of substituted 2-amino-4-aryl-3-cyano-4*H*-pyrans are under extensive development because of biological activities of these compounds.[1—9] 2-Amino-4-aryl-3-cyano-4*H*-pyrans fused with fragments of coumarin[10] and substituted benzenes,[11] quinolines,[12] naphthalenes,[13] and pyrazolones,[14,15] which exhibit anticoagulating, antisclerotic, anticancer, and other practically important properties, are of the greatest interest as biologically active compounds.

Earlier, it was found that 1-acetyl-1,2-dihydro-3*H*-indol-3-one and 2*H*-benzo[*b*]thiophen-3-one react with 2-aryl(hetaryl)-1,1-dicyanoethylenes[16,17] to give 2-amino-4-aryl-3-cyano-substituted 4*H*-pyrano[3,2-*b*]indoles and 4*H*-benzo[*b*]thieno[3,2-*b*]pyrans. A one-step method for the synthesis of these compounds was developed. The method, which involves no preliminary synthesis or isolation of unsaturated nitriles, is based on three-component condensation of 1-acetyl-1,2-dihydro-3*H*-indol-3-one or 2*H*-benzo[*b*]thiophen-3-one with an aromatic aldehyde and malononitrile.[16,17] With the aim of further studying the effect of the heteroatom on the direction of condensation of five-membered benzoannelated heterocyclic ketones with 2-aryl(hetaryl)-1,1-dicyanoethylenes, we used in this reaction a new ketone, namely, 4,6-dimethyl-2*H*-thieno[2,3-*b*]pyridin-3-one (**1**).

The reactions of 4,6-dimethyl-2*H*-thieno[2,3-*b*]pyridin-3-one (**1**) with unsaturated nitriles **2** were carried out in boiling EtOH in the presence of triethylamine for a short period of time (Scheme 1).[17] Under these conditions, the reactions are highly regioselective and afford 2-amino-4-aryl-3-cyano-7,9-dimethyl-4*H*-pyrano[2´,3´:4,5]thieno[2,3-*b*]pyridines (**3**) in 53—85% yields (Table 1, method *A*).

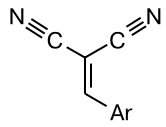
The scheme of this process can be represented as follows. Initially, the reaction of triethylamine with 3-oxothieno[2,3-*b*]pyridine **1** gives enolate anion **4**, which then is involved in a Michael reaction with arylmethylidenemalononitrile **2**. In basic media, Michael adduct **5** undergoes intramolecular cyclization into iminopyran **6** (Scheme 2). Subsequent tautomeric conversion yields 4*H*-pyrano[3´,2´:4,5]thieno[3,2-*b*]pyridines **3**.

Scheme 1



2,3: Ar = Ph (**a**), 4-FC_6H_4 (**b**), 4-ClC_6H_4 (**c**), 3-$NO_2C_6H_4$ (**d**), 4-$MeOCOC_6H_4$ (**e**)

The direction of the reaction depends on the nature of a substituent in arylmethylidenemalononitrile **2**. For instance, the reaction easily occurs for nonsubstituted phenylmethylidenemalononitrile **2a** (Ar = Ph) and its substituted analogs containing an acceptor substituent such as a nitro group (compound **2d**) or a halogen atom (compounds **2b,c**). Arylmethylidenemalononitriles **2f,g**



2f,g

Ar = 4-$MeOC_6H_4$ (**f**), 3,4-$(MeO)_2C_6H_3$ (**g**)

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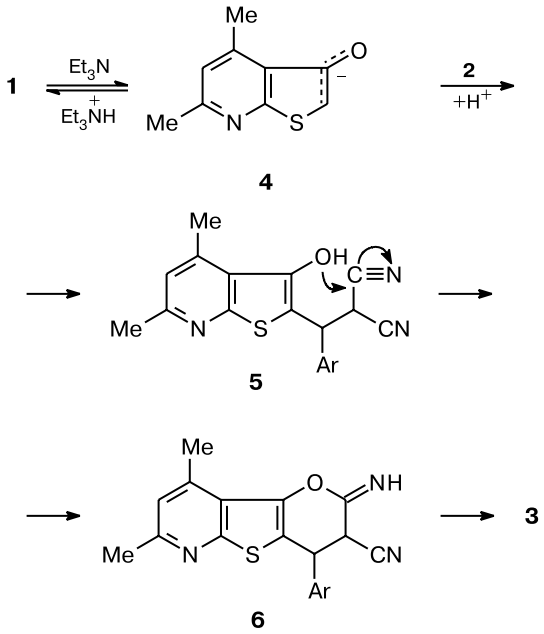
Table 1. Physicochemical characteristics of compounds **3**

Compound **3**	Ar	Yield (%) (method)	M.p. /°C	Found / Calculated (%)				Molecular formula	MS, m/z ($[M]^+$)
				C	H	N	S		
a	Ph	68 (*A*), 59 (*B*)	256—258	68.51 / 68.45	4.31 / 4.53	12.45 / 12.60	9.52 / 9.62	$C_{19}H_{15}N_3OS$	333
b	4-FC_6H_4	85 (*A*), 76 (*B*)	289—291	64.81 / 64.94	3.89 / 4.02	12.10 / 11.96	9.00 / 9.13	$C_{19}H_{14}FN_3OS$	351
c	4-ClC_6H_4	87 (*A*), 78 (*B*)	280—282	62.67 / 62.04	4.13 / 3.84	11.15 / 11.42	8.78 / 8.72	$C_{19}H_{14}ClN_3OS$	368
d	3-$NO_2C_6H_4$	74 (*A*), 65 (*B*)	245—247	59.93 / 60.31	3.59 / 3.73	14.71 / 14.81	8.37 / 8.47	$C_{19}H_{14}N_4O_3S$	378
e	4-$MeOCOC_6H_4$	60 (*A*), 72 (*B*)	283—285	63.82 / 64.43	4.43 / 4.38	10.94 / 10.73	8.15 / 8.19	$C_{21}H_{17}N_3O_3S$	391
f	3-FC_6H_4	57 (*B*)	273—275	64.34 / 64.94	3.75 / 4.02	11.75 / 11.96	9.01 / 9.13	$C_{19}H_{14}FN_3OS$	351
g	2-$CF_3C_6H_4$	61 (*B*)	245—247	59.83 / 59.84	3.81 / 3.52	10.23 / 10.47	7.85 / 7.99	$C_{20}H_{14}F_3N_3OS$	401
h	2-ClC_6H_4	61 (*B*)	294—296	62.37 / 62.04	3.57 / 3.84	11.20 / 11.42	8.80 / 8.72	$C_{19}H_{14}ClN_3OS$	368
i	4-BrC_6H_4	69 (*B*)	275—277	55.02 / 55.35	3.57 / 3.42	10.55 / 10.19	7.65 / 7.78	$C_{19}H_{14}BrN_3OS$	411, 413
j	3-C_5H_4N	72 (*B*)	>300	64.82 / 64.65	4.43 / 4.22	16.29 / 16.75	9.51 / 9.59	$C_{18}H_{14}N_4OS$	334
k	2-C_4H_3S	53 (*B*)	228—230	59.82 / 60.15	3.43 / 3.86	12.64 / 12.38	18.85 / 18.89	$C_{17}H_{13}N_3OS_2$	339

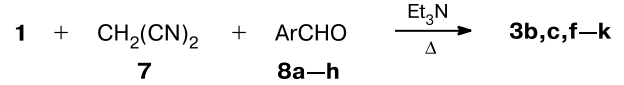
Scheme 2



containing electron-donor substituents (one or two alkoxy groups) do not enter into the reaction. Probably, this is due to a reduced electrophilicity of the C_β atom of an unsaturated nitrile because of the presence of the electron-releasing substituents.

4*H*-Pyrano[3′,2′:4,5]thieno[3,2-*b*]pyridines **3** can be obtained by a simpler method (method *B*) in one step by three-component condensation of 3-oxothieno[2,3-*b*]pyridine **1**, malononitrile **7**, and a corresponding aldehyde **8** (Scheme 3). In this case, the preliminary synthesis of arylmethylidenemalononitriles **2** is unnecessary. Earlier, three-component condensation was already used to synthesize substituted 2-amino-4*H*-pyrans. Condensation of aldehydes, malononitrile, and ethyl acetoacetate afforded the corresponding 2-amino-4-aryl-3-cyano-5-ethoxycarbonyl-6-methyl-4*H*-pyrans.[7]

Scheme 3

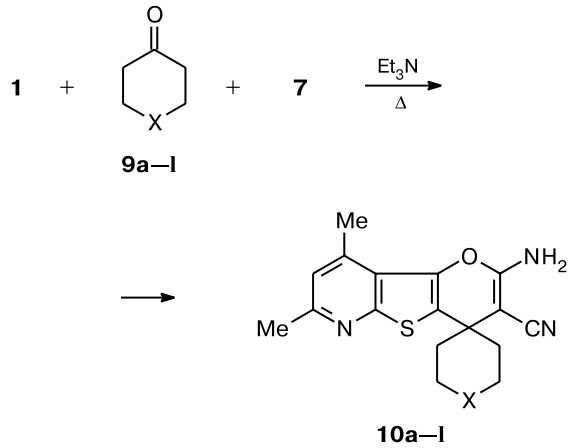


3: Ar = 4-FC_6H_4 (**b**), 4-ClC_6H_4 (**c**), 3-FC_6H_4 (**f**), 2-$CF_3C_6H_4$ (**g**), 2-ClC_6H_4 (**h**), 4-BrC_6H_4 (**i**), 3-C_5H_4N (**j**), 2-C_4H_3S (**k**)

8: Ar = 4-FC_6H_4 (**a**), 4-ClC_6H_4 (**b**), 3-FC_6H_4 (**c**), 2-$CF_3C_6H_4$ (**d**), 2-ClC_6H_4 (**e**), 4-BrC_6H_4 (**f**), 3-C_5H_4N (**g**), 2-C_4H_3S (**h**)

The reaction was carried out in boiling EtOH in the presence of triethylamine for a short period of time. Under these conditions, the reaction proceeds regioselectively to give pyrans **3**. Although method *B* provides lower yields of the target products than method *A* (see Table 1), yet the former yields, taking into account the prelimi-

Scheme 4



9,10: X = CH_2 (**a**), CH—Me (**b**), CH—Et (**c**), CH—Bu^t (**d**), N—Me (**e**), N—Et (**f**), N—Pr^n (**g**), N—Pr^i (**h**), N—Ac (**i**), N—COOEt (**j**), N—$COOBu^t$ (**k**), N—$COOCH_2Ph$ (**l**)

nary synthesis of unsaturated nitriles **2**, are higher by ~11—17%.

With the goal of synthesizing difficultly accessible spiro heterocycles, we used ketones (cyclohexanone and piperidone derivatives **9**) instead of aromatic aldehydes in these reactions (Scheme 4). It was found that heating of 3-oxothieno[2,3-*b*]pyridine **1** with ketone **9** and malononitrile **7** in EtOH (see also Refs. 18, 19) affords, in one step, compounds **10** in 25—86% yields (Table 2). Spiro compounds **10** are representatives of a novel class of heterocyclic systems, namely, piperidine-4-spiro- and cyclohexane-4-spiro-(4*H*)pyrano[2′,3′:4,5]thieno[2,3-*b*]pyridines. The suggested reaction mechanism is similar to that shown in Scheme 2.

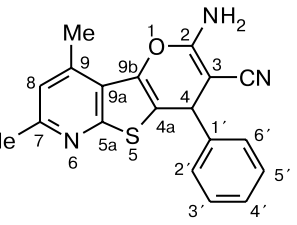
The structures of compounds **3** and **10** were confirmed by data from instrumental analytical methods (Tables 1—4).

The IR spectra of compounds **3** show absorption bands of the deformation and stretching vibrations of the amino group at 1647—1670 and 3239—3481 cm^{-1}, respectively. Absorption bands of the conjugated cyano group of the enaminonitrile fragment in the pyran ring appear at 2198—2240 cm^{-1}. The pattern is analogous for benzoannelated 2-amino-4*H*-pyrans.[20,21] In the IR spectra of spiropyrans **10**, absorption bands of the deformation and stretching vibrations of the amino group appear at 1604—1656 and 3116—3460 cm^{-1}, while absorption bands of the conjugated cyano group of the enaminonitrile fragment in the pyran ring appears at 2180—2208 cm^{-1}. The IR spectra of compounds **10i—l** contain an absorption band at 1636—1694 cm^{-1} for the carbonyl group of the acylamide fragment.

Table 2. Physicochemical characteristics of compounds **10**

Compound **10**	X	Yield (%)	M.p. /°C	Found / Calculated (%)				Molecular formula	MS, *m/z* ([M]$^+$)
				C	H	N	S		
a	CH_2	73	226—228	66.18 / 66.43	6.13 / 5.88	13.16 / 12.91	9.40 / 9.85	$C_{18}H_{19}N_3OS$	325
b	CH—Me	51	208—210	67.10 / 67.23	6.37 / 6.24	12.46 / 12.38	9.44 / 9.45	$C_{19}H_{21}N_3OS$	339
c	CH—Et	81	196—198	67.83 / 67.96	6.71 / 6.56	11.97 / 11.87	9.10 / 9.07	$C_{20}H_{23}N_3OS$	353
d	CH—Bu^t	75	217—219	69.31 / 69.26	6.60 / 7.13	11.42 / 11.01	8.66 / 8.40	$C_{22}H_{27}N_3OS$	381
e	N—Me	71	205—207	63.82 / 63.50	5.74 / 5.92	16.62 / 16.46	9.45 / 9.42	$C_{18}H_{20}N_4OS$	340
f	N—Et	86	175—177	64.41 / 64.38	6.40 / 6.26	15.91 / 15.81	9.04 / 9.05	$C_{19}H_{22}N_4OS$	354
g	N—Pr^n	80	165—167	65.07 / 65.19	6.71 / 6.56	15.31 / 15.20	8.72 / 8.70	$C_{20}H_{24}N_4OS$	368
h	N—Pr^i	64	151—153	65.07 / 65.19	6.74 / 6.56	15.29 / 15.20	8.72 / 8.70	$C_{20}H_{24}N_4OS$	368
i	N—Ac	56	259—261	61.82 / 61.94	5.59 / 5.47	15.31 / 15.21	8.72 / 8.70	$C_{19}H_{20}N_4O_2S$	368
j	N—COOEt	65	219—221	60.52 / 60.28	5.68 / 5.56	14.49 / 14.06	8.46 / 8.05	$C_{20}H_{22}N_4O_3S$	398
k	N—COO—Bu^t	63	209—211	61.52 / 61.95	6.02 / 6.14	12.97 / 13.14	7.49 / 7.52	$C_{22}H_{26}N_4O_2S$	426
l	N—$COOCH_2Ph$	25	145—147	65.27 / 65.20	5.10 / 5.25	12.32 / 12.17	6.82 / 6.96	$C_{25}H_{24}N_4O_3S$	460

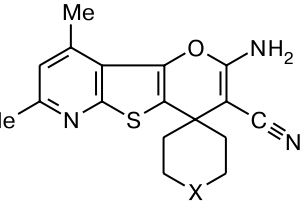
Table 3. Spectral characteristics of compounds **3**



Compound **3**	^{1}H NMR, δ						IR, ν/cm^{-1}		
	$H_3C(9)$ (s, 3 H)	$H_3C(7)$ (s, 3 H)	H(4) (s, 1 H)	NH_2 (br.s, 2 H)	H(8) (s, 1 H)	Ar	δ(NH_2)	CN	NH_2
a	2.50	2.70	4.90	6.82	7.00	7.25—7.49 (m, 5 H)	1660	2212	3300, 3352
b	2.50	2.70	4.95	6.90	7.01	7.10—7.16, 7.30—7.35 (both m, 2 H each)	1660	2210	3244, 3325, 3476
c	2.50	2.70	4.95	6.95	7.01	7.35 (m, 4 H)	1660	2210	3240, 3330, 3474
d	2.50	2.70	5.03	6.88	7.04	7.40—7.67, 8.45—8.60 (both m, 2 H each)	1670	2240	3260, 3358, 3410
e*	2.50	2.70	5.02	6.96	7.03	3.88 (s, 3 H, 4′-OMe); 7.41, 7.99 (both d, 2 H each, J = 8.17 Hz)	1662	2210	3278, 3325, 3398
f	2.50	2.70	4.98	6.85	7.01	7.07 (s, 1 H); 7.15—7.41 (m, 3 H)	1662	2210	3245, 3332, 3481
g	2.50	2.70	5.22	6.98	7.01	7.41—7.71 (m, 4 H)	1657	2218	3247, 3321, 3468
h	2.50	2.70	5.42	6.99	7.01	7.25—7.48 (m, 4 H)	1647	2213	3334, 3460, 3618
i	2.50	2.70	4.95	6.90	7.05	7.15—7.31, 7.40—7.60 (both m, 2 H each)	1661	2215	3239, 3320, 3448
j	2.50	2.70	5.01	6.87	7.01	7.30—7.38, 7.60—7.70 (both m, 1 H each); 8.45—8.58 (m, 2 H)	1658	2198	3292, 3370
k	2.50	2.70	3.88	6.50		6.90 (br.s, 2 H); 7.32 (br.s, 1 H)	1665	2214	3250, 3330, 3378

* ν(CO) = 1710 cm^{-1}.

Table 4. Spectral characteristics of compounds **10**



Compound **10**	^{1}H NMR, δ					IR, ν/cm^{-1}			
	X(—CH_2—)	$H_3C(9)$	$H_3C(7)$ (s, 3 H)	NH_2 (br.s, 2 H)	H(8) (s, 1 H)	δ(NH_2)	CO	CN	NH_2
a	1.40—1.90 (m, 10 H, CH_2)	2.52 (s, 3 H)	2.70	6.50	6.99	1654	—	2184	3172, 3270, 3312
b	1.05 (s, 3 H, CHC$\underline{H}_3$); 1.45—1.80 (m, 5 H, C$\underline{H}$CH_3, CH_2); 1.90 (s, 4 H, CH_2)	2.55 (s, 3 H)	2.65	6.49	6.99	1656	—	2184	3124, 3302, 3348
c	0.97 (t, 3 H, $CH_2C\underline{H}_3$, J = 1.0 Hz); 1.35—1.90 (m, 11 H, CH_2, C$\underline{H}_2$CH_3, CH_2)	2.50 (s, 3 H)	2.69	6.49	6.99	1644	—	2188	3176, 3312, 3418

(*to be continued*)

Table 4 (*continued*)

Compound **10**	^{1}H NMR, δ					IR, ν/cm^{-1}			
	X(—CH_2—)	H_3C(9)	H_3C(7) (s, 3 H)	NH_2 (br.s, 2 H)	H(8) (s, 1 H)	δ(NH_2)	CO	CN	NH_2
d*	0.90—1.00 (m, 10 H, CH, $(CH_3)_3C(4')$); 1.10—1.20 (m, 1 H, CH); 1.55—2.03 (m, 6 H, CH); 2.10—2.20 (m, 1 H, CH)	2.50 (s, 3 H)	2.61	6.51	6.99	1646	—	2196	3380, 3316, 3460
e**	1.80—1.90, 2.05—2.20 (both m, 2 H each, CH_2); 2.45—2.60 (m, 8 H, $H_3C(1')$, CH, H_3C(9)); 2.68—2.82 (m, 2 H, CH)		2.65	6.60	7.00	1640	—	2184	3168, 3304, 3400
f	1.10 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.2 Hz); 1.80—1.92, 2.02—2.18 (both m, 2 H each, CH_2); 2.40—2.60 (m, 7 H, 1′-$C\underline{H}_2CH_3$, CH,H_3C (9)); 2.80—2.90 (m, 2 H, CH)		2.65	6.70	7.00	1638	—	2180	3116, 3296, 3412
g	0.95 (t, 3 H, $C\underline{H}_3CH_2CH_2$, J = 6.9 Hz); 1.55 (q, 2 H, $CH_2C\underline{H}_2CH_3$); 1.80—1.91, 2.00—2.15, 2.30—2.42 (all m, 2 H each, CH); 2.50—2.70 (m, 5 H, $C\underline{H}_2CH_2CH_3$, H_3C (9)); 2.79—2.90 (m, 2 H, CH)		2.65	6.62	6.99	1648	—	2196	3198, 3344
h	1.05 (d, 6 H, $C\underline{H}_3CHCH_3$); 1.80—1.91, 2.00—2.11 (both m, 2 H each, CH); 2.50—2.58 (s, 5 H, CH, H_3C (9)); 2.70—2.87 (m, 3 H, $CH_3C\underline{H}CH_3$, CH)		2.62	6.65	7.00	1654	—	2184	3180, 3320
i	1.80—2.01 (m, 4 H, CH); 2.06 (s, 3 H, 1′-$COCH_3$); 3.20—3.38 (m, 1 H, CH); 3.48—3.68, 3.78—3.92 (both m, 1 H each, CH); 4.11—4.30 (m, 1 H, CH)	2.50 (s, 3 H)	2.62	7.00	7.10	1604	1636	2184	3160, 3304, 3388
j	1.30 (t, 3 H, $OCH_2C\underline{H}_3$, J = 7.1 Hz); 1.85—2.05 (m, 4 H, CH); 3.35—3.50 (m, 4 H, CH); 4.10 (2 H, q, $OC\underline{H}_2CH_3$)	2.51 (s, 3 H)	2.65	6.82	7.00	1642	1680	2188	3176, 3284, 3456
k***	1.48 (s, 9 H, $(C\underline{H}_3)_3C(4')$); 1.81—2.09 (m, 4 H, CH); 3.35—3.50, 3.87—4.00 (both m, 2 H each, CH)	2.51 (s, 3 H)	2.65	6.75	7.00	1654	1686	2188	3216, 3324, 3380
l	1.85—2.10 (m, 4 H, CH); 3.50 (br.s, 2 H, CH); 3.95—4.10 (m, 2 H, CH); 5.10 (s, 2 H, $COOC\underline{H}_2C_6H_5$); 7.28 (s, 5 H, $COOCH_2C_6\underline{H}_5$)	2.52 (s, 3 H)	2.68	6.84	7.01	1656	1694	2208	3212, 3328, 3424

The ratio of axial/equatorial atoms is *1 : 1, **1 : 7, and ***1 : 5.

The ^{1}H NMR data confirmed the structures of pyrans **3**. Thus, along with signals for the aryl fragment at δ_H 6.90—8.60, the ^{1}H NMR spectrum shows a signal characteristic of C(4)H as a singlet at δ_H 3.88—5.42. Signals for the protons of an amino group appear as a broadened singlet at δ_H 6.82—6.96.

The ^{1}H NMR spectra of compounds **10** contain signals characteristic of the protons of an amino group as a broadened singlet at δ_H 6.49—7.01 and of the protons of a hydrogenated heterocycle at δ_H 0.90—4.10.

Experimental

Melting points of the products obtained were determined on a Kofler stage. IR spectra were recorded on a Specord IR-75 instrument (KBr pellets). ^{1}H NMR spectra were recorded on a

Bruker M-300 instrument. The course of the reaction was monitored and the purity of the products was checked by TLC on Silufol UV-254 plates.

Compound **1** was prepared as described earlier.[22]

Synthesis of compounds 3a—e (general procedure). ***A.*** Triethylamine (0.3 mL) was added at 40—60 °C to a solution of equimolar amounts of compound **1** (1.79 g, 0.01 mol) and arylmethylidenemalononitrile **2a—e** (0.01 mol) in 30 mL of EtOH. The reaction mixture was kept at 20 °C for 24 h, and the precipitate that formed was filtered off, washed with EtOH and hexane, and recrystallized from MeCN (see Tables 1, 3).

Synthesis of compound 3b,c,d,f—k (general procedure). ***B.*** Triethylamine (0.3 mL) was added at 40—60 °C to a solution of equimolar amounts of compound **1** (1.79 g, 0.01 mol), aldehyde **8a—h** (0.01 mol), and malononitrile **7** (0.01 mol) in 30 mL of EtOH. The reaction mixture was kept at 20 °C for 24 h, and the precipitate that formed was filtered off, washed with EtOH and hexane, and recrystallized from MeCN (see Tables 1, 3).

Synthesis of compounds 10a—l (general procedure). Triethylamine (0.3 mL) was added at 40—60 °C to a solution of equimolar amounts of compound **1** (1.79 g, 0.01 mol), ketone **9a—l** (0.01 mol), and malononitrile **7** (0.01 mol) in 30 mL of EtOH. The reaction mixture was kept at 20 °C for 24 h, and the precipitate that formed was filtered off, washed with EtOH and hexane, and recrystallized from MeCN (see Tables 2, 4).

References

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