SYNTHESIS OF 2-ARYLFURO[3,2-*c*]PYRIDINES AND THEIR DERIVATIVES

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A series of 2-arylfuro[3,2-*c*]pyridines was synthesized. 3-(5-Aryl-2-furyl)propenoic acids **1a–1h** were converted to the acid azides **2a–2h**, which in turn were cyclized to give 2-arylfuro[3,2-*c*]pyridine-4(5*H*)-ones **4a–4h** by heating in Dowtherm. The pyridones **4a–4f** were aromatized with phosphorus oxychloride to the 2-aryl-4-chlorofuro[3,2-*c*]pyridines **5a–5f**, which were reduced with zinc and acetic acid to the title compounds **6a–6f**. Reacted with phosphorus(V) sulfide, the pyridones **4a–4f** yielded the corresponding thiones **7a–7f**.

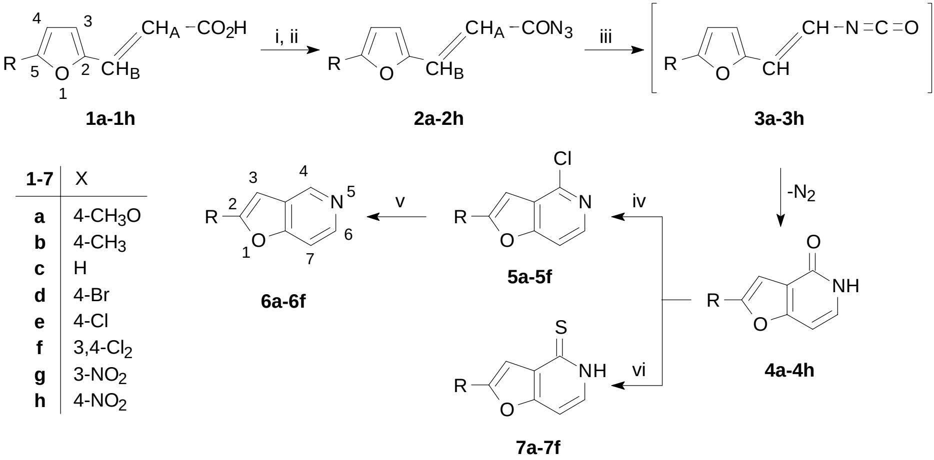
Key words: Furopyridines; [1,5]-Prototropic rearrangement.

The fusion of a π-excessive furan ring to a π-deficient pyridine nucleus gives rise to six isomeric furopyridines; the members of all types are known[1]. Approaches to the synthesis of furo[3,2-*c*]pyridines are based either on furan ring formation starting from substituted pyridines[2–5] or on pyridine ring formation starting from suitable substituted furans[6–11].

Previously we reported on the synthesis and reactions of 2-nitrophenylfuro[3,2-*c*]pyridine[12], 2,3-dimethylfuro[3,2-*c*]-pyridine[13], and pyrrolo[2′,3′:4,5]furo[3,2-*c*]pyridines[14]. Reactions[12,13,15–19], spectral[20], and biological properties[21] of the furo[3,2-*c*]pyridine derivatives have been studied.

The present paper describes efficient syntheses of some representatives of the title ring system (Scheme 1) developed in relation to our previous efforts aimed at the preparation of fused *O*-, *N*-heterocycles. Condensation of 5-aryl-2-furancarbaldehydes with malonic acid afforded 3-(5-aryl-2-furyl)propenoic acids **1a–1h**. Since the formerly described[22] Perkin synthesis gave these compounds in low yields, we applied Doebner reaction conditions in the synthesis resulting in very high yields of the products. The acids **1a–1h** were reacted with ethyl chloroformate and triethylamine in acetone to give mixed acid anhydrides, which were converted to the acid azides **2a–2h** with (*E*)-configuration at the double bond (see values for *J*(A,B) in Table I). The azides **2a–2h** were heated in Dowtherm at 240 °C, which facilitated the Curtius rearrangement to the corresponding non-isolated isocyanates **3a–3h**. The isocyanates apparently undewent isomerization from (*E*) to (*Z*) during the electrocyclic reaction leading to **4a–4h** in the presence of tributylamine, which served to transfer a proton in the [1,5]-prototropic rearrangement (Scheme 2).

The structures of the 2-arylfuro[3,2-*c*]pyridine-4(5*H*)-ones **4a–4h** were confirmed by spectroscopic data (Table II). Their IR spectra exhibit lactam ν(C=O) vibrations



In formulae **1-7** R = X-substituted phenyl

i, $(C_2H_5)_3N$, $ClCO_2C_2H_5$; ii, NaN_3; iii, $(nC_4H_9)_3N$, dowtherm; iv, $POCl_3$; v, Zn, CH_3CO_2H; vi, P_4S_{10}

SCHEME 1

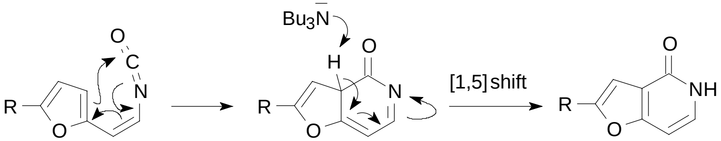
TABLE I

^{1}H NMR data ($CDCl_3$) of compounds **2**

Compound	H-A(d)	H-B(d)	H-3(d)	H-4(d)	Arom.(m)	*J*(A,B)	*J*(3,4)
2a[a]	6.30	7.47	6.76	6.62	6.91–7.72	15.3	3.5
2b[a]	6.34	7.29	6.77	6.69	7.18–7.77	15.4	3.5
2c	6.35	7.38	6.77	6.63	7.42–7.94	15.4	3.5
2d	6.36	7.35	6.76	6.65	7.53–7.81	15.3	3.5
2e	6.65	7.48	6.76[b]		7.29–7.71	15.7	–
2f	6.37	7.48	6.77[b]		7.49–7.76	15.3	–
2g	6.40	7.46	6.75	6.63	7.60–8.54	15.4	3.4
2h	6.42	7.48	6.77	6.67	7.89–8.32	15.3	3.5

[a] Other signals: **2a**: 3.85 s, 3 H (CH_3O); **2b:** 2.38 s, 3 H (CH_3). [b] s.

at 1 664–1 680 cm^{-1} and ν(NH) vibrations at 3 098–3 105 cm^{-1}. The 2-arylfuro[3,2-*c*]pyridine-4(5*H*)-ones **4a–4f** were transformed to 2-aryl-4-chlorofuro[3,2-*c*]pyridines **5a–5f** by refluxing with phosphorus oxychloride. The ^1H NMR spectra (Table II) of compounds **5a–5f** showed long-range coupling between H-3 and H-7, which is consistent with the so-called "zig-zag" path hypothesis and has also been reported for furo[3,2-*c*]pyridine[2,7]. Reduction of the compounds **5a–5f** with zinc and acetic acid afforded the 2-arylfuro[3,2-*c*]pyridines **6a–6f**. The position of the pyridine ring protons in the compounds **5a–5f** and **6a–6f** (Table II) was downfield shifted. As concerns the effect of the 2-aryl group on the chemical shift on pyridine carbons (see compound **6a**) it is small as compared with the values of the parent furo[3,2-*c*]pyridine (ref.[20]), in contrast to the effect on the furanoid carbons C-2 and C-3 which is high. This fact indicates that the electronic effect of the pyridine ring in this fused system upon C-2 and C-3 is low.



SCHEME 2

Reaction of the selected pyridones **4a–4f** with phosphorus(V) sulfide afforded the corresponding thiones **7a–7f**. The IR spectra of these compounds showed an absorption band within the region of 3 170 to 3 110 cm^{-1} ($\nu(C–H_{arom})$). The vibrational wavenumbers of the N–H and C=S bonds lay within the ranges of 3 210–3 250 cm^{-1} and 1 538–1 591 cm^{-1}, respectively. ^1H NMR spectral data of compounds **7** are summarized in Table II. The thioamide group in compounds **7c** and **7d** in dimethyl sulfoxide solutions showed a tendency to tautomerize into the thiol form, owing to which the –SH group can be directly substituted by a hydrazino group. An example of this reaction has been presented in ref.[12].

Mass spectra of the final 2-arylfuro[3,2-*c*]pyridines **6a–6f** displayed molecular peaks ($M^{+\bullet}$) of 100% relative intensity (Table III).

EXPERIMENTAL

Melting points were determined on a Kofler stage and are uncorrected. ^1H NMR spectra were recorded on a Tesla BS 587 (80 MHz) and ^{13}C NMR spectra on a Varian VXR-300 (300 MHz) spectrometer in $CDCl_3$ or $(CD_3)_2SO$ with TMS and HMDS as internal standards. Chemical shifts are given in ppm (δ-scale), coupling constants *J* in Hz. IR spectra were measured on an FTIR PU 9802/8 (Philips) spectrophotometer using the KBr technique ($\tilde{\nu}$ in cm^{-1}), UV spectra of ethanolic solutions (λ in nm, ε in $m^2\ mol^{-1}$) were recorded on a Specord UV-VIS M-40 (Zeiss, Jena) instrument. Mass spectra were taken on an MS 902-S instrument (AEI Manchester); direct inlet, ionizing electron energy

Table II
^{1}H NMR data ($(CD_3)_2SO$) of compounds **4–7**

Comp.	H-3 (s)	H-4 (s)	N-H (brs)	H-6 (d)	H-7 (d)	Arom. (m)	*J*(3,7)	*J*(6,7)	Other signals
4a	7.20	–	11.33	7.24	6.62	6.92–7.78	–	7.3	3.85 s, 3 H (CH_3O)
4b	7.87	–	11.38	7.24	6.62	7.16–7.73	–	7.1	2.4 s, 3 H (CH_3)
4c	7.37	–	11.40	7.26	6.55	7.30–7.90	–	7.1	–
4d	7.46	–	11.43	7.29	6.64[a]	7.53–7.81	0.6	7.2	–
4e	7.45	–	11.45	7.30	6.64	7.41–7.87	–	7.3	–
4f	7.58	–	11.49	7.31	6.65	7.58–8.04	–	7.1	–
4g	7.69	–	11.50	7.33	6.69	7.60–8.53	–	7.3	–
4h	7.75	–	11.61	7.35	6.68	7.97–8.32	–	7.3	–
5a	6.91[b]	–	–	8.21	7.36[a]	6.93–7.85	0.9	5.7	3.87 s, 3 H (CH_3O)
5b	7.02	–	–	8.23	[c]	7.26–7.82	–	5.7	2.42 s, 3 H (CH_3)
5c	7.08[b]	–	–	8.24	7.41[a]	7.42–7.94	0.9	5.7	–
5d	7.10	–	–	8.26	7.41	7.54–7.81	–	5.7	–
5e	7.01[b]	–	–	8.27	7.41[a]	7.40–7.87	0.8	5.7	–
5f	7.11[b]	–	–	8.29	7.41[a]	7.48–7.97	0.7	5.7	–
6a[d]	6.90	8.87	–	8.45	7.42	6.93–8.42		5.7	3.86 s, 3 H (CH_3O)
6b	7.00	8.90	–	8.47	7.45	7.23–7.81	–	5.7	2.41 s, 3 H (CH_3)
6c[e]	7.05[b]	8.91[b]	–	8.49	[c]	7.93–7.93	0.8	5.7	–
6d	7.05	8.92	–	8.50	7.44	7.54–7.81	–	5.7	–
6e	7.05	8.92	–	8.45	[c]	7.38–7.85	–	5.7	–
6f	7.08	8.93	–	8.52	7.46	7.51–7.94	–	5.7	–
7a	7.28	–	13.17	7.56	7.14	7.18–7.87	–	7.1	3.27 s, 3 H (CH_3O)
7b	6.78	–	12.60	7.02	6.54	6.49–7.21	–	7.1	2.30 s, 3 H (CH_3)
7c	7.47[b]	–	13.21	[c]	7.15[a]	7.35–7.94	0.6	7.0	–
7d	7.54	–	13.23	[c]	7.11	7.51–7.88	–	6.9	–
7e	7.51	–	13.21	[c]	7.12	7.40–8.05	–	6.5	–
7f	7.69	–	13.25	7.57	7.13	7.62–8.15	–	7.1	–

[a] dd. [b] d. [c] In the multiplet of aromatic protons. [d] ^{13}C NMR spectrum ($CDCl_3$): 160.60 (C-4′), 158.05 (C-7a), 157.09 (C-2), 144.05 (C-6), 143.48 (C-4), 126.86 (C-1′), 126.80 (C-2′), 122.26 (C-3a), 113.43 (C-3′), 106.77 (C-7), 97.35 (C-3), 55.41 (OCH_3). [e] *J*(4,7) = 0.8 Hz.

70 eV, trap current 100 μA, ion sorce temperature 160–220 °C. The starting 5-aryl-2-furancarbaldehydes were prepared following refs[23,24].

3-(5-Aryl-2-furyl)propenoic Acids **1a–1h**. General Procedure

A mixture of 5-aryl-2-furancarbaldehyde (10 mmol), malonic acid (1.04 g, 10 mmol) and pyridine (5 ml) was heated on a steam bath for 8 h. The reaction mixture was poured on crushed ice acidified with hydrochloric acid. The separated precipitate was filtered out. Characteristic data of the acids **1a–1e**, **1g** and **1h** are consistent with ref.[22].

3-[5-(3,4-Dichlorophenyl)-2-furyl]propenoic Acid (**1f**). 5-(3,4-Dichlorophenyl)-2-furancarbaldehyde afforded 95% of **1f**, m.p. 125–128 °C (80% aqueous ethanol). For $C_{13}H_8Cl_2O_3$ (283.1) calculated: 55.15% C, 2.85% H, 25.05% Cl; found: 55.28% C, 2.88% H, 25.15% Cl. ^{1}H NMR spectrum ($(CD_3)_2SO$): 12.24 brs, 1 H (CO_2H); 7.56–8.15 m, 3 H (arom.); 7.37 d, 1 H *J*(AB) = 15.6 (H-B); 7.22 d, 1 H *J*(3,4) = 3.5 (H-3); 6.98 d, 1 H (H-4); 6.37 d, 1 H (H-A). IR spectrum: 1 664 (C=O).

3-(5-Aryl-2-furyl)propenoyl Azides **2a–2h**. General Procedure

3-(5-Aryl-2-furyl)propenoic acid (8 mmol) dissolved in dry acetone (10 ml) was cooled to –10 °C, and triethylamine (0.9 g, 9.3 mmol) and ethyl chloroformate (1.13 g, 10 mmol) in dry acetone (1 ml) were added dropwise to the stirred system held at a temperature lower than 0 °C. The reaction mixture was stirred for another 30 min at the same temperature, and a solution of sodium azide (0.8 g, 12 mmol) in water (5 ml) was added while keeping the temperature below 0 °C. The formed yellow suspension was stirred for an additional hour and allowed to warm to room temperature. The mixture

TABLE III
Mass spectral data of compounds **6**

Compound	*m*/*z* (relative abundance)
6a	226 (18), 225 ($M^{+\bullet}$, 100), 210 (57), 182 (45), 127 (18), 126 (6.8), 112.5 (7.7), 101 (7.2), 77 (13.6), 75 (9.1), 63 (18.2), 62 (6.6)
6b	209 ($M^{+\bullet}$, 100), 208 (25), 180 (12.5), 152 (12.5), 139 (12.2), 127 (8), 15 (8.5), 104.5 (7.5), 91.5 (9), 91 (9.2), 76 (9.7), 65 (10.2), 63 (12.5), 51 (8.5)
6c	195 ($M^{+\bullet}$, 100), 166 (20), 140 (20), 139 (28), 114 (8.4), 97.5 (8), 77 (16), 63 (12), 51 (12)
6d	275 (100), 273 ($M^{+\bullet}$, 100), 195 (11), 167, (11.7), 166 (5.2), 140 (17.2), 139 (37.9), 138 (10.2), 137.5 (10.2), 136 (10.3), 120 (11.7), 113 (16.2), 97 (6.2), 87 (9.7), 83 (6.6), 76 (15.9), 75 (20.7), 69.5 (20.7), 69 (7.6), 63 (27.6), 62 (10.2), 51 (8.6), 50 (17.2), 39 (8.4)
6e	231 (36), 229 ($M^{+\bullet}$, 100), 195 (5.8), 173 (5.2), 166 (30), 140 (6.8), 139 (18), 14.5 (8), 113 (7), 111 (7.2), 75 (6), 63 (6), 62 (5.6), 50 (5.2), 39 (26)
6f	267 (13.9), 265 (66.7), 263 ($M^{+\bullet}$, 100), 202 (7.2), 200 (27.8), 175 (9.6), 164 (5), 147 (9.6), 137 (5.3), 132.5 (5), 131.5 (7.8), 109 (6.1), 87 (9.6), 80 (6.9), 75 (6.4), 74 (8.3), 62 (7.8)

TABLE IV
Yields and physical properties of compounds **2–5**

Compound	Yield, %	M.p., °C (solvent)	Formula (M.w.)	Calculated/Found % C	Calculated/Found % H	Calculated/Found % N	IR ν(C=O)
2a	88	101–102 (acetone)	$C_{14}H_{11}N_3O_3$ (269.3)	62.45 62.33	4.12 3.92	15.61 15.21	
2b	90	110–111 (acetone)	$C_{14}H_{11}N_3O_2$ (253.3)	66.40 66.32	4.38 4.22	16.59 16.12	
2c	90	106–108 (acetone)	$C_{13}H_9N_3O_2$ (239.1)	65.25 64.95	3.79 3.60	17.57 16.98	
2d	85	106–108 (acetone)	$C_{13}H_8BrN_3O_2$ (318.1)	49.08 48.62	2.53 2.43	13.21 12.93	
2e	96	105 (acetone)	$C_{13}H_8ClN_3O_2$ (273.7)	57.05 56.94	2.95 3.27	15.35 14.91	
2f	75	78–80 (acetone)	$C_{13}H_7Cl_2N_3O_2$ (308.1)	50.68 50.38	2.29 2.28	13.64 13.58	
2g	86	118–120 (acetone)	$C_{13}H_8N_4O_4$ (284.2)	54.94 54.56	2.84 2.73	19.71 19.65	
2h	90	126–127 (acetone)	$C_{13}H_8N_4O_4$ (284.2)	54.94 54.78	2.84 2.56	19.71 19.55	
4a	55	240–242 (ethanol)	$C_{14}H_{11}NO_3$ (241.3)	69.70 69.57	4.60 4.76	5.81 5.59	1 674
4b	63	272–274 (DMF)	$C_{14}H_{11}NO_2$ (225.3)	74.65 74.39	4.92 4.57	6.22 6.13	1 655
4c	55	258–259 (water)	$C_{13}H_9NO_2$ (211.2)	73.92 73.57	4.29 4.39	6.63 6.57	1 664
4d	59	318–320 (DMF)	$C_{13}H_8BrNO_2$ (290.11)	53.82 53.70	2.78 2.63	4.83 4.81	1 676
4e	89	304–306 (DMF)	$C_{13}H_8ClNO_2$ (245.7)	63.56 63.39	3.28 3.25	5.70 5.64	1 678

TABLE IV
(*Continued*)

Compound	Yield, %	M.p., °C (solvent)	Formula (M.w.)	Calculated/Found % C	% H	% N	IR ν(C=O)
4f	45	279–280	$C_{13}H_7Cl_2NO_2$	55.74	2.52	5.00	1 680
		(ethanol)	(280.1)	55.69	2.48	4.89	
4g	40	247–249	$C_{13}H_8N_2O_4$	60.94	3.15	10.93	1 678
		(DMF)	(352.2)	60.84	3.12	11.02	
4h	35	349–352	$C_{13}H_8N_2O_4$	60.94	3.15	10.93	1 664
		(DMF)	(352.2)	60.61	2.98	10.56	
5a	60	150–151	$C_{14}H_{10}ClNO_2$	64.75	3.88	5.39	
		(i-hexane)	(259.7)	64.39	3.71	5.51	
5b	55	142–144	$C_{14}H_{10}ClNO$	69.00	4.14	5.75	
		(ethanol)	(243.7)	68.75	4.31	5.90	
5c	75	96–98	$C_{13}H_8ClNO$	67.99	3.51	6.10	
		(ethanol)	(229.8)	67.80	3.43	6.12	
5d	64	203	$C_{13}H_7BrClNO$	50.60	2.29	4.54	
		(ethanol)	(308.6)	50.34	2.21	4.56	
5e	71	213	$C_{13}H_7Cl_2NO$	59.12	2.67	5.30	
		(ethanol)	(264.1)	58.94	2.52	5.28	
5f	75	212–214	$C_{13}H_6Cl_3NO$	52.30	2.03	4.69	
		(ethanol)	(298.6)	52.28	2.00	4.65	

was poured onto crushed ice and the yellow precipitate was filtered out and rinsed with water. Yields, m.p. and elemental analyses are given in Table IV.

2-Arylfuro[3,2-*c*]pyridin-4(5*H*)-ones **4a–4h**. General Procedure

A mixture of 3-(5-aryl-2-furyl)propenoyl azide (17 mmol), toluene (20 ml), Dowtherm (12 ml) and tributylamine (3.69 ml, 22.5 mmol) was heated for 45 min at 180–200 °C, during which toluene was allowed to distill off continuously. After cooling, diethyl ether was added, the precipitate formed was filtered out and rinsed with diethyl ether. The characteristic data are given in Table IV.

2-Aryl-4-chlorofuro[3,2-*c*]pyridines **5a–5f**. General Procedure

2-Arylfuro[3,2-*c*]pyridin-4(5*H*)-one (20 mmol) was refluxed in phosphorus oxychloride (10 ml) for 4 h. $POCl_3$ was distilled off at a reduced pressure, and ice was added to the residue. The mixture was then

TABLE V
Yields and physical properties of compounds **6** and **7**

Compound	Yield %	M.p., °C (solvent)	Formula (M.w.)	Calculated/Found			UV λ(log ε)	IR ν(C=S)
				% C	% H	% N		
6a	62	129–130	$C_{14}H_{11}NO_2$	74.65	4.92	6.22	306 (3.49)	–
		(i-hexane)	(225.3)	74.36	4.85	6.08	206 (3.47)	
6b	52	115–116	$C_{14}H_{11}NO$	80.36	5.30	6.69	302 (3.39)	–
		(i-hexane)	(209.3)	80.43	5.30	6.67	204 (3.56)	
6c	67	115–116	$C_{13}H_9NO$	79.98	4.65	7.17	296 (3.49)	–
		(ethanol	(195.2)	79.84	4.62	7.11	201 (3.56)	
6d	56	167–168	$C_{13}H_8BrNO$	56.96	2.94	5.11	302 (3.61	
		(ethanol)	(274.1)	57.41	3.03	5.09	203 (3.54)	
6e	69	113–114	$C_{13}H_8ClNO$	67.99	3.51	6.10	301 (3.56)	–
		(ethanol	(229.7)	67.94	3.65	5.90	202 (3.54)	
6f	70	153–158	$C_{13}H_7Cl_2NO$	59.12	2.67	5.30	302 (3.52)	–
		(ethanol)	(264.1	59.20	2.66	5.25	208 (3.45)	
7a	85	257–259	$C_{14}H_{11}NO_2S$[a]	65.35	4.31	5.44	–	1 538
		(ethanol)	(257.3)	65.29	4.21	5.56		1 238
7b	80	281–282	$C_{14}H_{11}NOS$[a]	69.68	4.59	5.80	–	1 591
		(dioxane)	(241.3)	69.48	4.51	5.76		1 240
7c	92	244–246	$C_{13}H_9NOS$[a]	68.70	3.99	6.16	–	1 587
		(ethanol)	(227.3)	68.43	3.76	5.95		1 234
7d	74	297–299	$C_{13}H_8BrNOS$[a]	51.00	2.63	4.57	–	1 587
		(ethanol)	(306.2)	50.74	2.62	4.38		1 238
7e	52	257–258	$C_{13}H_8ClNOS$[a]	59.66	3.08	5.35	–	1 570
		(ethanol)	(261.7)	59.71	2.83	5.24		1 240
7f	86	274–276	$C_{13}H_7Cl_2NOS$[a]	52.72	2.38	4.73	–	1 570
		(dioxane)	(296.2)	52.34	2.32	4.48		1 240

[a] % S; **7a** calculated: 12.46, found: 12.34; **7b** calculated: 13.29, found: 12.99; **7c** calculated: 14.11, found: 13.73; **7d** calculated: 10.47, found: 10.28; **7e** calculated: 12.25, found: 11.97; **7f** calculated: 10.82, found: 10.61.

made basic with dilute aqueous ammonia. The precipitate obtained was filtered out, rinsed with water, dried and crystallized. The characteristic data are given in Table IV.

2-Arylfuro[3,2-*c*]pyridines **6a–6f**. General Procedure

A mixture of 2-aryl-4-chlorofuro[3,2-*c*]pyridine (5.5 mmol), Zn (pulverized, 2.2 g) and glacial acetic acid (10 ml) was refluxed for 8 h. After cooling, the solid was removed by filtration and the filtrate was evaporated to dryness at a reduced pressure. Dilute NaOH solution was added to the residue, and the alkaline mixture was extracted with chloroform. The characteristic data are given in Table V.

2-Arylfuro[3,2-*c*]pyridin-4(5*H*)-thiones **7a–7f**. General Procedure

A mixture of 2-arylfuro[3,2-*c*]pyridin-4(5*H*)-one **4a–4f** (10 mmol) and phosphorus(V) sulfide (2.22 g, 5 mmol) was refluxed in pyridine (10 ml) for 4 h while stirred, and poured into hot water (30 ml). The separated precipitate was filtered out, dried and crystallized. The characteristic data of compounds **7** are given in Table V.

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