

Reactions of Methyl 2-Formylfuro[3,2-*b*]pyrrole-5-carboxylates

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A series of 5-methoxycarbonyl-4-R-furo[3,2-*b*]pyrrole-2-carbaldehyde 2,6-R¹,R²-phenylhydrazones and 5-methoxycarbonyl-4-R-furo[3,2-*b*]pyrrole-2-carbaldehyde *N,N*-dimethylhydrazones were prepared from methyl 2-formyl-4-R-furo[3,2-*b*]pyrrole-5-carboxylates. By the reaction of the latter with hydroxylammonium chloride in acetic anhydride in the presence of pyridine methyl 2-cyano-4-R-furo[3,2-*b*]pyrrole-5-carboxylates were obtained. The reaction of these compounds with sodium azide and ammonium chloride in dimethylformamide led to methyl 2-(5'-tetrazolyl)-4-R-furo[3,2-*b*]pyrrole-5-carboxylates. The reaction of methyl 2-formyl-4-R-furo[3,2-*b*]pyrrole-5-carboxylates with malononitrile afforded 5-methoxycarbonyl-4-R-furo[3,2-*b*]pyrrol-2-ylidenemalononitriles, with methyl cyanoacetate methyl 2-cyano-3-[(5-methoxycarbonyl-4-R-furo[3,2-*b*]pyrrol)-2-yl]propenoates and with 2-furylacetonitrile 2-(2-furyl)-3-[(5-methoxycarbonyl-4-R-furo[3,2-*b*]pyrrol)-2-yl]acrylonitriles.

In continuation of our program aimed at developing efficient syntheses of fused nitrogen-containing heterocycles, we here report on the study of the utilization of substituted furo[3,2-*b*]-type aldehydes in the synthesis.

Our previous paper [1] presents the formylation of methyl furo[3,2-*b*]pyrrole-5-carboxylate and its 4-methyl or 4-benzyl derivatives under conditions of Vilsmeier reaction. In that reaction analogously as in the case of ethyl 4*H*-furo[3,2-*b*]pyrrole-5-carboxylate and its 4-methyl [2] or 4-benzyl derivative [3] methyl 2-formyl-4-R-furo[3,2-*b*]pyrrole-5-carboxylates (*Ia—Ic*) were obtained.

By the reaction of substituted aldehydes of the furo[3,2-*b*]pyrrole type (*Ia—Ic*) with 2,6-R¹,R²-phenylhydrazinium chlorides and sodium acetate in methanol—water ($\varphi_r = 3:1$) hydrazones (*IIa—IIIi*) were obtained. *N,N*-Dimethylhydrazones *IIIa—IIIc* started from *unsym*-dimethylhydrazine and mentioned aldehydes in refluxing toluene, with a catalytic amount of 4-methylbenzenesulfonic acid.

The reaction of *Ia—Ic* with hydroxylammonium chloride in acetic anhydride in the presence of pyridine at 90 °C gave corresponding cyano-substituted compounds *IVa—IVc*. The reaction of *IVa—IVc* with sodium azide and ammonium chloride in dimethylformamide led to *Va—Vc*.

The reaction of compounds *Ia—Ic* with malonic acid dinitrile afforded *VIa—VIc*, with methyl cyanoacetate *VIIa—VIIc*, and with 2-furylacetonitrile *VIIIa—VIIIc*.

Characteristic data, UV and IR spectra of synthesized compounds are listed in Table 1. The structure of the studied compounds has been confirmed by

¹H NMR spectra as well (Tables 2 and 3). The signals of furo[3,2-*b*]pyrrole protons H-3 and H-6 display doublets resulting from a long-range coupling $^5J_{3,6} = 0.8$ Hz or broad signals with unresolved coupling constants. For compounds, where R = H, H-6 signal is split by the NH proton with coupling constant $^4J_{4,6} = 1.7$ Hz. The double bond signal H-7 appears as singlet for all compounds.

EXPERIMENTAL

Melting points were determined on a Kofler hot-stage apparatus. ¹H NMR spectra were recorded on Tesla BS 587 spectrometer (80 MHz), *IIa—IIIi* and *IIIa—IIIc* were measured in (CD₃)₂CO and the other compounds in DMSO-*d*₆. The UV spectra were measured on a M-40 (Zeiss, Jena) spectrophotometer in methanol, $c = 10^{-4}$ mol dm⁻³ (see Table 1). The IR spectra were taken on a FTIR PU 9802/25 (Philips) spectrophotometer using KBr technique (0.5 mg in 300 mg KBr).

The starting compounds were prepared according to the literature: 2,6-R¹,R²-phenylhydrazinium chlorides [4, 5] (the corresponding 2,6-R¹,R²-phenylhydrazines were liberated from their crystalline salts with sodium hydroxide), methyl 2-formyl-4-R-furo[3,2-*b*]pyrrole-5-carboxylates [1], 2-furylacetonitrile [6].

5-Methoxycarbonyl-4-R-furo[3,2-*b*]pyrrole-2-carbaldehyde 2,6-R¹,R²-Phenylhydrazone (*IIa—IIIi*)

A solution of 2,6-R¹,R²-phenylhydrazinium chloride (10 mmol) and sodium acetate (0.86 g; 10 mmol) in

Table 1. Characterization and Spectral Data of Synthesized Compounds^a

Compound	Formula M_r	$w_i(\text{calc.})/\%$ $w_i(\text{found})/\%$			M.p./°C Yield/%	λ_{max} nm	UV $\log(\epsilon/(\text{m}^2 \text{mol}^{-1}))$	IR, $\bar{\nu}/\text{cm}^{-1}$	
		C	H	N				$\nu(\text{C=O})$	$\nu(\text{CN})$
<i>IIa</i>	$\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}_3$	65.58	5.50	13.50	174—177	362	3.75	1692	
	311.3	65.42	5.38	13.44	82				
<i>IIb</i>	$\text{C}_{18}\text{H}_{19}\text{N}_3\text{O}_3$	66.45	5.89	12.91	149—152	362	3.75	1695	
	325.4	66.52	5.79	12.84	78				
<i>IIc</i>	$\text{C}_{19}\text{H}_{21}\text{N}_3\text{O}_3$	67.24	6.24	12.38	149—151	362	3.76	1689	
	339.4	67.44	6.36	12.54	75				
<i>IId</i>	$\text{C}_{18}\text{H}_{19}\text{N}_3\text{O}_3$	66.45	5.89	12.91	158—159	364	3.72	1682	
	325.4	66.66	5.82	12.81	84				
<i>IIe</i>	$\text{C}_{19}\text{H}_{21}\text{N}_3\text{O}_3$	67.24	6.24	12.38	105—106	364	3.70	1682	
	339.4	67.36	6.42	12.44	80				
<i>IIf</i>	$\text{C}_{20}\text{H}_{23}\text{N}_3\text{O}_3$	67.97	6.56	11.89	104—107	364	3.72	1682	
	353.4	67.88	6.46	11.92	77				
<i>IIg</i>	$\text{C}_{24}\text{H}_{23}\text{N}_3\text{O}_3$	71.80	5.77	10.47	124—126	365	3.69	1688	
	401.5	71.68	5.90	10.37	76				
<i>IIh</i>	$\text{C}_{25}\text{H}_{25}\text{N}_3\text{O}_3$	72.27	6.06	10.11	89—93	365	3.70	1688	
	415.5	72.47	6.16	10.02	74				
<i>IIi</i>	$\text{C}_{26}\text{H}_{27}\text{N}_3\text{O}_3$	72.71	6.34	9.78	116—120	365	<i>b</i>	1701	
	429.5	72.77	6.38	9.68	72				
<i>IIIa</i>	$\text{C}_{11}\text{H}_{13}\text{N}_3\text{O}_3$	56.16	5.57	17.86	199—201	357	3.75	1694	
	235.2	56.10	5.70	17.78	72				
<i>IIIb</i>	$\text{C}_{12}\text{H}_{15}\text{N}_3\text{O}_3$	57.82	6.07	16.86	86—89	359	3.79	1690	
	249.3	57.70	6.17	16.92	70				
<i>IIIc</i>	$\text{C}_{18}\text{H}_{19}\text{N}_3\text{O}_3$	66.45	5.89	12.91	110—113	361	3.71	1696	
	325.4	66.55	5.79	12.77	69				
<i>IVa</i>	$\text{C}_9\text{H}_6\text{N}_2\text{O}_3$	56.85	3.18	14.73	227—231	315	3.64	1695	2220
	190.2	56.75	3.28	14.78	84	307			
<i>IVb</i>	$\text{C}_{10}\text{H}_8\text{N}_2\text{O}_3$	58.82	3.95	13.72	194—195	317	3.67	1684	2220
	204.2	58.76	3.92	13.70	82	310			
<i>IVc</i>	$\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_3$	68.57	4.32	9.99	116—118	318	3.63	1714	2214
	280.3	68.47	4.22	10.02	76	309			
<i>Va</i>	$\text{C}_9\text{H}_7\text{N}_5\text{O}_3$	46.36	3.03	30.03	263—265 ^c	320	3.74	1695	
	233.2	46.48	3.22	30.16	74	329			
<i>Vb</i>	$\text{C}_{10}\text{H}_9\text{N}_5\text{O}_3$	48.59	3.67	28.33	244—246	319	3.69	1703	
	247.2	48.44	3.56	28.50	72	330			
<i>Vc</i>	$\text{C}_{16}\text{H}_{13}\text{N}_5\text{O}_3$	59.44	4.05	21.66	232—235	320	3.65	1705	
	323.3	59.36	4.12	21.62	70	331			
<i>VIa</i>	$\text{C}_{12}\text{H}_7\text{N}_3\text{O}_3$	59.75	2.93	17.42	237—243	417	3.78	1703	2228
	241.2	59.68	2.90	17.36	88				
<i>VIb</i>	$\text{C}_{13}\text{H}_9\text{N}_3\text{O}_3$	61.18	3.55	16.46	241—245	418	3.73	1707	2220
	255.2	61.32	3.44	16.40	86				
<i>VIc</i>	$\text{C}_{19}\text{H}_{13}\text{N}_3\text{O}_3$	68.88	3.95	12.68	168—170	417	3.82	1709	2224
	331.3	68.66	3.90	12.72	82				
<i>VIIa</i>	$\text{C}_{13}\text{H}_{10}\text{N}_2\text{O}_5$	56.94	3.68	10.22	258—263	411	<i>b</i>	1694	2228
	274.2	56.88	3.72	10.33	82				
<i>VIIb</i>	$\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_5$	58.33	4.20	9.72	251—253	411	<i>b</i>	1703	2222
	288.3	58.16	4.12	9.68	86				
<i>VIIc</i>	$\text{C}_{20}\text{H}_{16}\text{N}_2\text{O}_5$	65.93	4.43	7.69	181—183	410	3.69	1711	2220
	364.3	65.78	4.42	7.88	78				
<i>VIIIa</i>	$\text{C}_{15}\text{H}_{10}\text{N}_2\text{O}_4$	63.83	3.57	9.92	230—232	417	3.84	1692	2224
	282.3	63.72	3.42	9.98	76				
<i>VIIIb</i>	$\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_4$	64.86	4.08	9.45	227—228	419	<i>b</i>	1701	2212
	296.3	64.88	4.28	9.40	74				
<i>VIIIc</i>	$\text{C}_{22}\text{H}_{16}\text{N}_2\text{O}_4$	70.96	4.33	7.52	157—159	422	3.75	1684	2218
	372.4	70.86	4.44	7.66	72				

a) Compounds were crystallized: *IIi*, *IIIa*—*IIIb* from *i*-hexane, all others from methanol; b) saturated solution; c) decomposition.

Table 2. ¹H NMR Data of Compounds *Iia*–*Iii* and *IIIa*–*IIIc*^a

Compound	δ_i				
	H-3	H-6	H-7	OCH ₃	Other signals, $J_{H,H}$
<i>Iia</i> ^b	6.56 bs	6.70 dd	7.50 s	3.80 s	R: 10.50 (bs, NH); R ¹ , R ² : 2.30 (s, CH ₃); 8.58 (bs, NH), $J_{3,6} = 0.8$ Hz, $J_{4,6} = 1.7$ Hz
<i>Iib</i> ^b	6.56 bs	6.70 dd	7.47 s	3.80 s	R: 10.50 (bs, NH); R ¹ : 2.31 (s, CH ₃); R ² : 2.72 (q, CH ₂), 1.19 (t, CH ₃); 8.56 (bs, NH), $J_{3,6} = 0.8$ Hz, $J_{4,6} = 1.7$ Hz
<i>Iic</i> ^b	6.54 bs	6.69 dd	7.44 s	3.80 s	R: 10.50 (bs, NH); R ¹ , R ² : 2.70 (q, CH ₂), 1.19 (t, CH ₃); 8.54 (bs, NH), $J_{3,6} = 0.8$ Hz, $J_{4,6} = 1.7$ Hz
<i>Iid</i> ^b	6.70 bs	6.70 bs	7.50 s	3.78 s	R: 3.95 (s, N—CH ₃); R ¹ , R ² : 2.32 (s, CH ₃); 8.62 (bs, NH)
<i>Iie</i> ^b	6.67 s	6.70 s	7.47 s	3.78 s	R: 3.94 (s, N—CH ₃); R ¹ : 2.31 (s, CH ₃); R ² : 2.72 (q, CH ₂), 1.19 (t, CH ₃); 8.58 (bs, NH)
<i>Iif</i> ^b	6.65 s	6.70 s	7.44 s	3.78 s	R: 3.94 (s, N—CH ₃); R ¹ , R ² : 2.70 (q, CH ₂), 1.19 (t, CH ₃); 8.56 (bs, NH)
<i>Iig</i> ^b	6.53 bs	6.80 d	7.44 s	3.81 s	R: 5.69 (s, CH ₂), 7.26 (m, H _{arom}); R ¹ , R ² : 2.30 (s, CH ₃); $J_{3,6} = 0.8$ Hz
<i>Iih</i> ^b	6.51 bs	6.80 d	7.41 s	3.78 s	R: 5.69 (s, CH ₂), 7.26 (m, H _{arom}); R ¹ : 2.28 (s, CH ₃); R ² : 2.70 (q, CH ₂), 1.17 (t, CH ₃); 8.59 (bs, NH), $J_{3,6} = 0.8$ Hz
<i>Iii</i> ^b	6.48 bs	6.79 d	7.38 s	3.77 s	R: 5.68 (s, CH ₂), 7.26 (m, H _{arom}); R ¹ , R ² : 2.68 (q, CH ₂), 1.17 (t, CH ₃); 8.50 (bs, NH), $J_{3,6} = 0.8$ Hz
<i>IIIa</i>	6.48 s	6.70 bs	7.15 s	3.80 s	R: 10.52 (bs, NH); 2.97 (s, N(CH ₃) ₂)
<i>IIIb</i>	6.57 s	6.70 s	7.13 s	3.78 s	R: 3.94 (s, N—CH ₃); 2.97 (s, N(CH ₃) ₂)
<i>IIIc</i>	6.43 s	6.80 s	7.09 s	3.77 s	R: 5.70 (s, CH ₂), 7.26 (m, H _{arom}); 2.97 (s, N(CH ₃) ₂)

a) Measured in (CD₃)₂CO; b) 7.00–7.15 (m, H-3', H-4', H-5' of H_{arom}).

water (5 cm³) and methanol (15 cm³) was treated with a hot solution of methyl 2-formyl-4-R-furo[3,2-*b*]pyrrole-5-carboxylate in methanol at room temperature. Stirring was continued for 10 min and fine yellow crystals began to separate from solution. The reaction mixture was allowed to stand for a few hours in refrigerator and the crystals were filtered off.

5-Methoxycarbonyl-4-R-furo[3,2-*b*]pyrrole-2-carbaldehyde Dimethylhydrazone (*IIIa*–*IIIc*)

A stirred solution of methyl formyl-4-R-furo[3,2-*b*]pyrrole-5-carboxylate (10 mmol) in toluene (10 cm³) containing a catalytic amount of 4-methylbenzenesulfonic acid (5 mg) was treated carefully with *N,N*-dimethylhydrazine (0.60 g; 10 mmol) in toluene (3 cm³). The solution was then refluxed for 2 h and water formed during the reaction was removed in the Dean–Stark trap. The solvent was removed under reduced pressure and the residue was crystallized.

Methyl 2-Cyano-4-R-furo[3,2-*b*]pyrrole-5-carboxylate (*IVa*–*IVc*)

To the mixture of methyl formyl-4-R-furo[3,2-*b*]pyrrole-5-carboxylate (*Ia*–*Ic*) (10 mmol), pyridine (8 cm³),

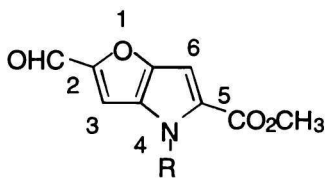
and hydroxylammonium chloride (1.2 g; 17 mmol) acetic anhydride (5.5 cm³) was added under stirring at 95 °C. The reaction mixture was kept at 85–95 °C for 2 h, cooled and poured on ice. The separated precipitate was filtered off and crystallized.

Methyl 2-(5'-Tetrazolyl)-4-R-furo[3,2-*b*]pyrrole-5-carboxylate (*Va*–*Vc*)

A stirred mixture of methyl 2-cyano-4-R-furo[3,2-*b*]pyrrole-5-carboxylate (*IVa*–*IVc*) (10 mmol), sodium azide (0.72 g; 12 mmol), ammonium chloride (0.64 g; 12 mmol), and dimethylformamide (13 cm³) was heated at 100 °C for 4 h. The solvent was distilled off *in vacuo*, the residue was dissolved in water, the solution was acidified with hydrochloric acid and the precipitate was filtered off and crystallized.

5-Methoxycarbonyl-4-R-furo[3,2-*b*]pyrrole-2-ylidenemalononitrile (*VIa*–*VIc*)

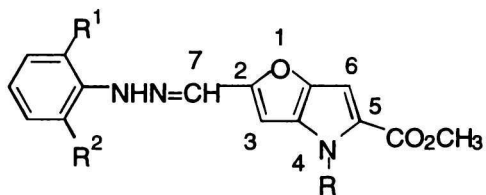
To a stirred solution of methyl 2-formyl-4-R-furo[3,2-*b*]pyrrole-5-carboxylate (*Ia*–*Ic*) (10 mmol) and malononitrile (0.78 g; 10 mmol) in methanol (20 cm³) sodium methoxide (5 drops of 10% methanolic solution) was added. The solid thus formed was filtered off, washed with cold methanol and crystallized.



I

R

- a H
- b CH₃
- c CH₂C₆H₅

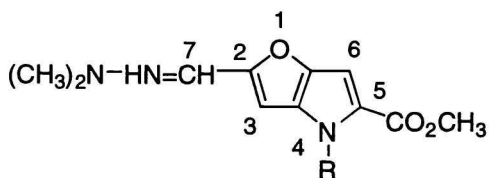


II

R

R¹R²

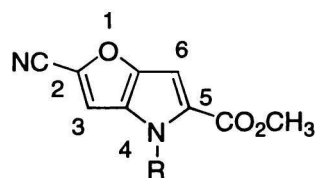
- | | | | |
|---|---|-------------------------------|-------------------------------|
| a | H | CH ₃ | CH ₃ |
| b | H | CH ₃ | C ₂ H ₅ |
| c | H | C ₂ H ₅ | C ₂ H ₅ |
| d | CH ₃ | CH ₃ | CH ₃ |
| e | CH ₃ | CH ₃ | C ₂ H ₅ |
| f | CH ₃ | C ₂ H ₅ | C ₂ H ₅ |
| g | CH ₂ C ₆ H ₅ | CH ₃ | CH ₃ |
| h | CH ₂ C ₆ H ₅ | CH ₃ | C ₂ H ₅ |
| i | CH ₂ C ₆ H ₅ | C ₂ H ₅ | C ₂ H ₅ |



III

R

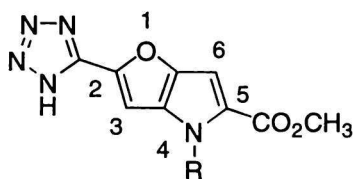
- a H
- b CH₃
- c CH₂C₆H₅



IV

R

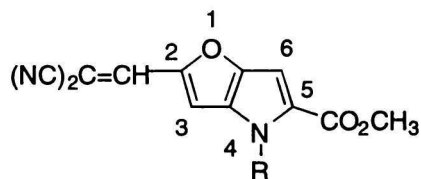
- a H
- b CH₃
- c CH₂C₆H₅



V

R

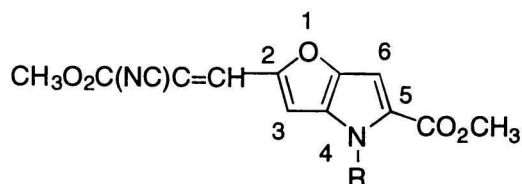
- a H
- b CH₃
- c CH₂C₆H₅



VI

R

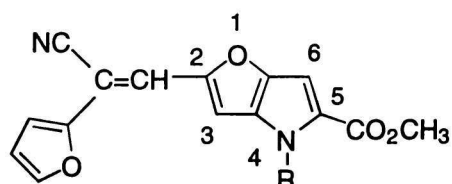
- a H
- b CH₃
- c CH₂C₆H₅



VII

R

- a H
- b CH₃
- c CH₂C₆H₅



VIII

R

- a H
- b CH₃
- c CH₂C₆H₅

Table 3. ^1H NMR Data of Compounds IV—VIII^a

Compound	δ_i				
	H-3	H-6	H-7	OCH ₃	Other signals, $J_{\text{H,H}}$
IVa	7.76 d	6.85 d	—	3.83 s	R: 12.20 (bs, NH)
IVb	7.91 d	6.83 d	—	3.81 s	R: 3.93 (s, N—CH ₃), $J_{3,6} = 0.8$ Hz
IVc	7.83 d	6.99 d	—	3.79 s	R: 5.67 (s, CH ₂), 7.00—7.38 (m, H _{arom}), $J_{3,6} = 0.8$ Hz
Va	7.30 s	6.87 bs	—	3.78 s	R: 12.00 (bs, NH)
Vb	7.56 s	6.87 s	—	3.74 s	R: 3.93 (s, N—CH ₃)
Vc	7.43 s	6.99 s	—	3.73 s	R: 5.68 (s, CH ₂), 7.10—7.38 (m, H _{arom})
VIa	7.43 d	6.88 d	8.11 s	3.80 s	R: 12.18 (s, NH), $J_{3,6} = 0.8$ Hz
VIb	7.62 d	6.90 d	8.22 s	3.82 s	R: 3.95 (s, N—CH ₃), $J_{3,6} = 0.8$ Hz
VIc	7.45 d	7.02 d	8.19 s	3.81 s	R: 5.72 (s, CH ₂), 7.00—7.35 (m, H _{arom}), $J_{3,6} = 0.8$ Hz
VIIa	7.57 d	6.87 d	8.10 s	3.82 s	R: 12.20 (s, NH); 3.84 (s, OCH ₃), $J_{3,6} = 0.8$ Hz
VIIb	7.68 d	6.87 d	8.07 s	3.82 s	R: 3.95 (s, N—CH ₃); 3.82 (s, OCH ₃), $J_{3,6} = 0.8$ Hz
VIIc	7.54 s	7.00 s	8.07 s	3.81 s	R: 5.71 (s, CH ₂), 7.00—7.40 (m, H _{arom}); 3.80 (s, OCH ₃), $J_{3,6} = 0.8$ Hz
VIIIa	7.22 s	6.88 bs	7.58 s	3.88 s	R: 12.00 (bs, NH); furan ring: 7.90 (d, H-5'), 6.82 (d, H-3'), 6.72 (dd, H-4'), $J_{3',4'} = 3.5$ Hz, $J_{4',5'} = 1.7$ Hz
VIIIb	7.36 bs	6.90 d	7.57 s	3.86 s	R: 4.00 (s, N—CH ₃); furan ring: 7.88 (d, H-5'), 6.83 (d, H-3'), 6.70 (dd, H-4'), $J_{3',4'} = 3.5$ Hz, $J_{4',5'} = 1.7$ Hz, $J_{3,6} = 0.8$ Hz
VIIIc	7.12 bs	6.93 d	7.47 s	3.86 s	R: 5.69 (s, CH ₂), 7.00—7.40 (m, H _{arom}); furan ring: 7.81 (d, H-5'), 6.77 (d, H-3'), 6.64 (dd, H-4'), $J_{3',4'} = 3.5$ Hz, $J_{4',5'} = 1.8$ Hz; $J_{3,6} = 0.8$ Hz

^a) Measured in (CD₃)₂SO.

Methyl 2-Cyano-3-[(5-methoxycarbonyl-4-R-furo[3,2-b]pyrrol)-2-yl]propenoate (VIIa—VIIc)

To a solution of Ia—Ic (10 mmol) and a freshly distilled methyl cyanoacetate (0.99 g; 10 mmol) in methanol (20 cm³) sodium methoxide (5 drops of 10 % methanolic solution) was added under stirring. After a short time fine crystals formed were filtered off, washed with cold methanol and crystallized.

2-(2-Furyl)-3-[(5-methoxycarbonyl-4-R-furo[3,2-b]pyrrol)-2-yl]acrylonitrile (VIIIa—VIIIc)

To a solution of Ia—Ic (10 mmol) and a freshly distilled 2-furylacetonitrile (1.07 g; 10 mmol) in methanol (20 cm³) sodium methoxide (5 drops of 10 % methanolic solution) was added under stirring. The reaction mixture was refluxed for 1 h. After cooling fine crystals formed were filtered off, washed with cold methanol and crystallized.

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