DERIVATIVES OF FURO[3,2-*b*]PYRROLE

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As a part of our program aimed at developing efficient syntheses of condensed nitrogen containing heterocycles, we have reported in the past[1,2] efficient syntheses of the title compounds. In continuation of this work we here report the syntheses of some representatives of title ring systems.

A number of methyl 2-substituted, 2,3-disubstituted-4*H*-furo[3,2-*b*]pyrrole-5-carboxylates (*V* – *VII*) and methyl 1*H*-benzo[*b*]furo[3,2-*b*]pyrrole-2-carboxylate (*VIII*) was prepared by the thermolysis of corresponding methyl 2-azido-3-(R^1,R^2-substituted-2-furyl)propenoates (*I* – *IV*). N-Methyl and N-benzyl derivatives *IX* – *XVI* were prepared at the phase-transfer catalysis conditions. Hydrolysis of some N-methyl and N-benzyl substituted esters furnished corresponding acids *XVII* – *XXII*. The hydrazides *XXIII* – *XXX* were obtained from corresponding esters and hydrazine hydrate. The preparation of 1-benzylbenzo[*b*]furo[3,2-*b*]pyrrole (*XXXI*) is described as well.

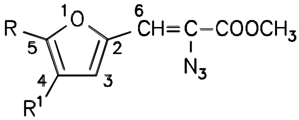
EXPERIMENTAL

Melting points were determined on a Kofler hot plate apparatus and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Tesla BS 587 (80 MHz for ^{1}H and 20.18 MHz for ^{13}C) spectrometer (TMS as internal standard). UV spectra were measured on a M-40 (Zeiss, Jena) spectrophotometer in ethanol (λ_{max}/log ε; λ_{max} in nm, ε in $m^2\ mol^{-1}$). The IR spectra were recorded on a FTIR PU 9802/25 (Philips) spectrophotometer using KBr technique (0.5 mg/300 mg KBr, ν given in cm^{-1}).

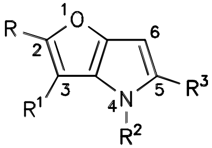
Characteristic data of synthesized compounds are given in Table I. The structure of the synthesized compounds has been confirmed by ^{1}H NMR spectroscopy (Tables II and III). IR and UV spectra are presented in Table IV. The ^{1}H NMR and ^{13}C NMR data of compounds *I* – *IV* are given in Experimental.

Compounds *I* – *IV* exist as Z isomers. This fact was established from ^{13}C NMR spectra of the compound *II* on the basis of the value of vicinal coupling constant ^3J(CO, H-6) = 5.0 Hz. The measured coupling constant is comparable to those of analogous compounds, as are ethyl 2-azido-3-(5-X-2-furyl)propenoates and 2-azido-3-(4,5-dimethyl-2-furyl)propenoate, which were studied in our earlier papers[3,4].

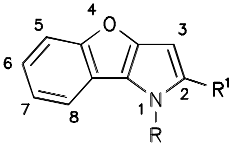
The following starting compounds were prepared: 4,5-dimethyl-2-furaldehyde according to ref.[5], 5-(4-chloro-2-methylphenoxy)-2-furancarbaldehyde according to ref.[6], benzo[*b*]furan-2-carbaldehyde according to ref.[7] and 1-acetylbenzo[*b*]furo[3,2-*b*]pyrrole according to ref.[8].



	R	R^1
I	CH_3	H
II	CH_3	CH_3
III	$4{-}Cl{-}2{-}CH_3C_6H_3O$	H
IV	$CH{=}CH{-}CH{=}CH$	



	R	R^1	R^2	R^3
V	CH_3	H	H	$COOCH_3$
VI	CH_3	CH_3	H	$COOCH_3$
VII	$4{-}Cl{-}2{-}CH_3C_6H_3O$	H	H	$COOCH_3$
IX	CH_3	H	CH_3	$COOCH_3$
X	CH_3	CH_3	CH_3	$COOCH_3$
XI	$4{-}Cl{-}2{-}CH_3C_6H_3O$	H	CH_3	$COOCH_3$
XIII	CH_3	H	$C_6H_5CH_2$	$COOCH_3$
XIV	CH_3	CH_3	$C_6H_5CH_2$	$COOCH_3$
XV	$4{-}Cl{-}2{-}CH_3C_6H_3O$	H	$C_6H_5CH_2$	$COOCH_3$
XVII	CH_3	H	CH_3	COOH
XVIII	CH_3	CH_3	CH_3	COOH
XIX	$4{-}Cl{-}2{-}CH_3C_6H_3O$	H	CH_3	COOH
XX	CH_3	H	$C_6H_5CH_2$	COOH
XXI	CH_3	CH_3	$C_6H_5CH_2$	COOH
XXIII	CH_3	CH_3	H	$CONHNH_2$
XXIV	CH_3	H	CH_3	$CONHNH_2$
XXV	CH_3	CH_3	CH_3	$CONHNH_2$
XXVI	$4{-}Cl{-}2{-}CH_3C_6H_3O$	H	CH_3	$CONHNH_2$
XXVII	CH_3	H	$C_6H_5CH_2$	$CONHNH_2$
XXVIIII	CH_3	CH_3	$C_6H_5CH_2$	$CONHNH_2$
XXVIX	$4{-}Cl{-}2{-}CH_3C_6H_3O$	H	$C_6H_5CH_2$	$CONHNH_2$



	R	R^1
VIII	H	$COOCH_3$
XII	CH_3	$COOCH_3$
XVI	$C_6H_5CH_2$	$COOCH_3$
XXII	$C_6H_5CH_2$	COOH
XXX	$C_6H_5CH_2$	$CONHNH_2$
XXXI	$C_6H_5CH_2$	H

Methyl 2-Azido-3-(5-methyl-2-furyl)propenoate (*I*)

A solution of 5-methyl-2-furancarbaldehyde (2.2 g, 0.02 mol) and methyl azidoacetate (9.2 g, 0.08 mol) was added at 0 °C during 30 min to sodium metal (1.84 g, 0.08 mol) in methanol (60 ml). Stirring was continued for additional 60 min at temperature not exceeding 5 °C, the reaction mixture was then cooled to 0 °C, a solution of ammonium chloride (4.4 g, 0.08 mol) in water (10 ml) was added and poured in ice water. The separated precipitate was filtered off and crystallized. Yield 2.57 g (62%); m. p. 30 – 32 °C (methanol). For $C_9H_9N_3O_3$ (207.2) calculated: 52.17% C, 4.4% H, 20.28% N; found: 52.26% C, 4.40% H, 20.44% N. IR spectrum: 2 118 (N_3), 1 707 (C=O). UV spectrum: 343 (3.40). ^{1}H NMR spectrum ($CDCl_3$): 7.00 d, 1 H (H-3, $J_{3,4}$ = 3.2); 6.81 s, 1 H (H-6); 6.12 bd, 1 H (H-4); 3.86 s, 3 H (OCH_3); 2.33 bs, 3 H (CH_3). Using the same method the compounds *II* – *IV* were prepared:

Methyl 2-azido-3-(4,5-dimethyl-2-furyl)propenoate (II): Yield 66%; m. p. 51 – 55 °C (methanol). For $C_{10}H_{11}N_3O_3$ (221.2) calculated: 54.29% C, 5.01% H, 19.00% N; found: 53.99% C, 4.96% H, 19.02% N. IR spectrum: 2 124 (N_3), 1 713 (C=O). UV spectrum: 352 (3.45). ^{1}H NMR spectrum ($CDCl_3$): 6.92 s, 1 H (H-3); 6.78 s, 1 H (H-6); 3.86 s, 3 H (OCH_3); 2.24 bs, 3 H (C_5-CH_3); 1.97 bs, 3 H (C_4-CH_3). ^{13}C NMR spectrum ($CDCl_3$): 163.9 (CO), 150.5 (C-5), 146.8 (C-2), 120.6 (C-7), 119.3 (C-3), 117.9 (C-4), 114.3 (C-6), 52.6 (O-CH_3), 11.6 (C_5-CH_3), 9.8 (C_4-CH_3), ^3J(CO, H-6) = 5.0 Hz.

Methyl 2-azido-3-[5-(4-chloro-2-methylphenoxy)-2-furyl]propenoate (III): Yield 54%; m. p. 47 – 49 °C (methanol). For $C_{15}H_{12}ClN_3O_4$ (333.7) calculated: 53.99% C, 3.68% H, 12.59% N; found: 53.82% C, 3.41% H, 12.68% N. IR spectrum: 2 126 (N_3), 1 711 (C=O). UV spectrum: 350 (3.45). ^{1}H NMR spectrum ($CDCl_3$): 7.04 d, 1 H (H-3, $J_{3,4}$ = 3.5); 6.72 s, 1 H (H-6); 6.80 – 7.28 m, 3 H (H arom.); 5.46 d, 1 H (H-4); 3.86 s, 3 H (OCH_3); 2.27 s, 3 H (CH_3).

Methyl 2-azido-3-(2-benzo[b]furyl)propenoate (IV): Yield 68%; m. p. 107 – 110 °C (methanol). For $C_{12}H_9N_3O_3$ (243.2) calculated: 59.26% C, 3.73% H, 17.28% N; found: 59.16% C, 3.68% H, 17.16% N. IR spectrum: 2 116 (N_3), 1 715 (C=O). UV spectrum: 347 (3.61). ^{1}H NMR spectrum ($CDCl_3$): 7.45 s, 1 H (H-3); 6.94 s, 1 H (H-6); 7.10 – 7.60 m, 4 H (H arom.); 3.91 s, 3 H (OCH_3).

TABLE I
Characteristic data of synthesized compounds *V* – *XXXI*

Compound	Formula (M.w.)	M.p., °C (Yield, %)	Calculated/Found		
			% C	% H	% N
V	$C_9H_9NO_3$ (179.2)	135 – 137 (67)	60.33 60.12	5.06 5.10	7.82 7.68
VI	$C_{10}H_{11}NO_3$ (193.2)	230 – 231 (72)	62.17 62.28	5.74 5.72	7.25 7.46
VII	$C_{15}H_{12}ClNO_4$ (305.7)	134 – 135 (36)	58.93 58.82	3.96 3.78	4.58 4.40
VIII	$C_{12}H_9NO_3$ (215.2)	203 – 204 (76)	66.97 66.92	4.22 4.16	6.51 6.38
IX	$C_{10}H_{11}NO_3$ (193.2)	30 – 32 (69)	62.17 62.32	5.44 5.67	7.25 7.34
X	$C_{11}H_{13}NO_3$ (207.2)	107 – 108 (73)	63.75 63.58	6.32 6.56	6.76 6.82
XI	$C_{16}H_{14}ClNO_4$ (319.7)	97 – 98 (46)	60.10 59.96	4.71 4.76	4.38 4.52
XII	$C_{13}H_{11}NO_3$ (229.2)	114 – 115 (72)	68.11 68.32	4.84 4.76	6.11 6.02
XIII	$C_{16}H_{15}NO_3$ (269.3)	109 – 111 (56)	71.36 71.44	5.61 5.72	5.20 5.38
XIV	$C_{17}H_{17}NO_3$ (283.3)	112 – 115 (58)	72.06 72.06	6.05 6.15	4.94 5.02
XV	$C_{22}H_{18}ClNO_4$ (395.8)	77 – 80 (39)	66.75 66.45	4.58 4.56	3.54 3.46
XVI	$C_{19}H_{15}NO_3$ (305.3)	124 – 125 (62)	74.74 74.82	4.95 5.02	4.59 4.70
XVII	$C_9H_9NO_3$ (179.2)	161 – 164 (64)	60.33 60.56	5.06 5.12	7.82 7.94

TABLE I
(*Continued*)

Compound	Formula (M.w.)	M.p., °C (Yield, %)	Calculated/Found		
			% C	% H	% N
XVIII	$C_{10}H_{11}NO_3$ (193.2)	182 – 184 (61)	62.17 62.12	5.74 5.58	7.25 7.21
XIX	$C_{15}H_{12}ClNO_4$ (305.7)	151 – 155 (42)	58.93 58.88	3.96 3.73	4.58 4.42
XX	$C_{15}H_{13}NO_3$ (255.3)	192 – 195 (64)	70.58 70.46	5.13 5.16	5.49 5.42
XXI	$C_{16}H_{15}NO_3$ (269.3)	150 – 156 (66)	71.36 71.45	5.61 5.62	5.20 5.34
XXII	$C_{18}H_{13}NO_3$ (291.5)	169 – 173 (60)	74.21 74.18	4.50 4.56	4.81 4.75
XXIII	$C_9H_{11}N_3O_2$ (193.2)	253 – 255 (72)	55.95 55.87	5.74 5.86	21.75 21.55
XXIV	$C_9H_{11}N_3O_2$ (193.2)	164 – 165 (69)	55.95 55.75	5.74 5.66	21.75 21.86
XXV	$C_{10}H_{13}N_3O_2$ (207.2)	215 – 220 (59)	57.96 57.88	6.32 6.56	20.28 20.56
XXVI	$C_{15}H_{14}ClN_3O_3$ (319.7)	167 – 169 (48)	56.35 56.46	4.41 4.56	13.14 13.34
XXVII	$C_{15}H_{15}N_3O_2$ (269.3)	143 – 145 (56)	66.90 66.88	5.61 5.58	15.60 15.46
XXVIII	$C_{16}H_{17}N_3O_2$ (283.3)	172 – 176 (62)	67.83 67.88	6.05 6.12	14.83 14.97
XXIX	$C_{21}H_{18}ClN_3O_3$ (395.8)	167 – 169 (42)	63.72 63.67	4.58 4.56	10.62 10.80
XXX	$C_{18}H_{15}N_3O_2$ (305.3)	197 – 199 (56)	70.81 70.76	4.95 4.90	13.76 13.66
XXXI	$C_{17}H_{13}NO$ (247.3)	71 – 72 (32)	82.57 82.50	5.30 5.45	5.66 5.44

TABLE II
^1H NMR spectra (δ, ppm) of furo[3,2-*b*]pyrrole derivatives

Compound	H-3[d]	H-6	R	R^1	R^2	R^3
V[a]	6.21 m	6.60 m	2.38 d (CH_3)	–	10.39 bs (NH)	3.79 s (OCH_3)
VI[a]	–	6.61 bs	2.35 m (CH_3)	2.11 m (CH_3)	10.45 bs (NH)	3.78 s (OCH_3)
VII[b]	5.87 d	6.68 m	6.90 – 7.44 m (H arom.), 2.30 s (CH_3)	–	11.70 bs (NH)	3.71 s (OCH_3)
IX[a]	6.31 m	6.68 bs	2.38 s (CH_3)	–	3.76 s (CH_3)	3.91 s (OCH_3)
X[a]	–	6.61 s	2.28 m (CH_3)	2.18 m (CH_3)	3.75 s (CH_3)	3.99 s (OCH_3)
XI[b]	6.04 bs	6.72 bs	6.90 – 7.44 m (H arom.), 2.22 s (CH_3)	–	3.69 s (CH_3)	3.82 s (OCH_3)
XIII[c]	5.79 m	6.89 d	2.34 d (CH_3)	–	5.60 s (CH_2), 7.00 – 7.37 m (C_6H_5)	3.78 s (OCH_3)
XIV[a]	–	6.75 s	2.27 m (CH_3)	2.03 m (CH_3)	5.79 s (CH_2), 6.95 – 7.40 m (C_6H_5)	3.72 s (OCH_3)
XV[b]	5.96 bs	6.81 bs	6.90 – 7.44 m (H arom.), 2.20 s (CH_3)	–	5.55 s (CH_2), 7.00 – 7.30 m (C_6H_5)	3.66 s (CH_3)
XVII[b]	6.35	6.61 bs	2.31 d (CH_3)	–	3.80 s (CH_3)	[e]
XVIII[b]	–	6.55 s	2.22 m (CH_3)	2.10 bs (CH_3)	3.89 s (CH_3)	[e]
XIX[b]	6.04 bs	6.68 bs	6.90 – 7.44 m (H arom.), 2.20 s (CH_3)	–	3.81 s (CH_3)	[e]
XX[b]	6.22 bs	6.70 bs	2.28 d (CH_3)	–	5.56 s (CH_2), 7.00 – 7.30 m (C_6H_5)	[e]
XXI[b]	–	6.68 s	2.20 bs (CH_3)	1.86 bs (CH_3)	5.68 s (CH_2), 7.00 – 7.30 m (C_6H_5)	11.90 bs (COOH)
XXIII[b]	–	6.59 d	2.20 bs (CH_3)	1.96 bs (CH_3)	11.10 bs (NH)	9.10 bs (NH), 4.24 bs (NH_2)
XXIV[b]	6.30 bs	6.62 bs	2.30 d (CH_3)	–	3.80 s (CH_3)	9.14 bs (NH), 4.20 bs (NH_2)
XXV[b]	–	6.54 s	2.20 bs (CH_3)	2.09 bs (CH_3)	3.88 s (CH_3)	9.09 bs (NH), 4.22 bs (NH_2)
XXVI[b]	6.07 bs	6.68 bs	6.90 – 7.40 m (H arom.), 2.23 s (CH_3)	–	3.82 s (CH_3)	9.17 bs (NH), 4.21 bs (NH_2)
XXVII[c]	5.80 bs	6.43 bs	2.34 d (CH_3)	–	5.61 s (CH_2), 7.00 – 7.37 m (C_6H_5)	3.60 bs (NH, NH_2)
XXVIII[b]	–	6.68 s	2.18 bs (CH_3)	1.86 bs (CH_3)	5.72 s (CH_2), 6.90 – 7.35 m (C_6H_5)	9.18 bs (NH), 4.20 bs (NH_2)
XXIX[b]	5.97 bs	6.76 bs	6.90 – 7.44 m (H arom.), 2.20 s (CH_3)	–	5.60 s (CH_2), 7.17 m (C_6H_5)	9.24 bs (NH), 4.20 bs (NH_2)

Solvents: [a] $(CD_3)_2CO$; [b] $(CD_3)_2SO$; [c] $CDCl_3$; [d] $J_{3,6}$ = 0.8 Hz or unresolved; [e] not observed.

TABLE III
^{1}H NMR spectra (δ, ppm) of benzo[*b*]furo[3,2-*b*]pyrrole derivatives

Compound	H-3	H-arom	R	R^1
VIII [a]	6.86 d	7.25 – 7.87 m	11.37 bs (NH)	3.87 s (OCH_3)
XII [b]	6.84 s	7.10 – 7.75 m	3.86 s (CH_3)	4.23 s (OCH_3)
XVI [c]	6.99 s	7.00 – 7.75 m	5.92 s (CH_2), 3.73 s (OCH_3)	
			m (C_6H_5) in H arom.	
XXII [c]	6.94 s	7.00 – 7.75 m	5.95 s (CH_2)	not observed
			m (C_6H_5) in H arom.	
XXX [c]	6.92 s	7.00 – 7.75 m	5.97 s (CH_2),	9.54 bs (NH)
			7.17 m (C_6H_5)	4.38 bs (NH_2)
XXXI [c]	6.19 d	7.00 – 7.60 m	5.31 s (CH_2),	6.79 d (H-2)
			7.26 m (C_6H_5)	*J*(2,3) = 3.1

Solvents: [a] $(CD_3)_2CO$; [b] $CDCl_3$; [c] $(CD_3)_2SO$.

TABLE IV
IR and UV spectra of synthesized compounds

Compound	ν(CO), cm^{-1}	λ_{max} (log ε), nm	Compound	ν(CO), cm^{-1}	λ_{max} (log ε), nm
V	1 665	306 (3.53), 251 (2.74)	*XIX*	1 647	304 (3.50)
VI	1 669	309 (3.50)	*XX*	1 646	305 (3.52)
VII	1 676	307 (3.56)	*XXI*	1 645	306 (3.46)
VIII	1 686	322 (3.67), 258 (3.18)	*XXII*	1 657	320 (3.55), 263 (3.12)
IX	1 680	307 (3.48)	*XXIII*	1 649	307 (3.45), 232 (2.84)
X	1 675	310 (3.52)	*XXIV*	1 628	303 (3.48)
XI	1 701	303 (3.58), 255 (2.87)	*XXV*	1 651	306 (3.62), 232 (3.03)
XII	1 692	323 (3.65), 263 (3.15)		1 626	
XIII	1 698	308 (3.50)	*XXVI*	1 634	304 (3.45)
XIV	1 694	311 (3.48)	*XXVII*	1 637	307 (3.47)
XV	1 703	309 (3.58), 256 (2.98)	*XXVIII*	1 640	306 (3.56)
XVI	1 694	324 (3.55), 263 (3.07)	*XXIX*	1 622	305 (3.41)
XVII	1 663	302 (3.44)	*XXX*	1 617	316 (3.41), 253 (3.08)
XVIII	1 645	306 (3.49)	*XXXI*	–	298 (3.12)

Methyl 2-R-3-R^1-4*H*-Furo[3,2-*b*]pyrrole-5-carboxylates (*V* – *VII*)

Methyl 2-azido-3-(R, R^1-substituted-2-furyl)propenoate (1 g) was dissolved in toluene (100 ml). The mixture was refluxed under stirring for 1 h, the solvent was evaporated in vacuo and the product was crystallized from methanol. Starting from *IV* analogously was prepared methyl 1*H*-benzo[*b*]furo-[3,2-*b*]pyrrole-2-carboxylate (*VIII*).

Methyl 2-R-3-R^1-4-Methylfuro[3,2-*b*]pyrrole-5-carboxylates (*IX* – *XII*)

A solution of sodium hydroxide (50%, 30 ml), methyl iodide (1.56 g, 11 mmol) and triethylbenzylammonium chloride (0.4 g) were added to a stirred solution of esters *IIa* – *IId* (10 mmol) in toluene (100 ml). The temperature was then raised to 65 °C and the mixture stirred for 4 h, diluted with water and the organic layer was separated. The aqueous layer was extracted with ether and combined with toluene solution, dried with sodium sulfate and the solvent was removed. The residue was crystallized from ethanol. Starting from *VIII* was made methyl 1-methylbenzo[*b*]furo[3,2-*b*]pyrrole-2-carboxylate (*XII*).

Methyl 2-R-3-R^1-4-Benzylfuro[3,2-*b*]pyrrole-5-carboxylates (*XIII* – *XV*)

A solution of sodium hydroxide (50%, 30 ml) and benzyl chloride (1.75 ml, 21 mmol) were added to stirred solution of the compounds *Ia* – *Id* (17 mmol) in toluene (30 ml). Finally triethylbenzylammonium chloride (1.2 g) was added and the mixture was kept stirred at 60 °C for 4 h. The cooled organic layer was separated, the aqueous one was three times extracted with toluene, the collected toluene layers were washed with water and dried with sodium sulfate. The product was crystallized after toluene has been distilled off under reduced pressure.

By the same way was prepared methyl- and benzylbenzo[*b*]furo[3,2-*b*]pyrrole-5-carboxylate (*XVI*).

2-R-3-R^1-4-R^2-Furo[3,2-*b*]pyrrole-5-carboxylic Acids (*XVII* – *XXI*)

Esters *IX* – *XI*, *XIII* – *XV* (0.01 mol) in ethanol (50 ml) and 5% sodium hydroxide (20 ml) were heated on steam bath for 1 h and concentrated to half of its original volume. The precipitate was dissolved in dilute ethanol (50%), acidified with hydrochloric acid 1 : 1 and poured into ice. The precipitate was filtered off and crystallized from methanol. Analogously was prepared 1-benzylbenzo[*b*]furo[3,2-*b*]pyrrole-2-carboxylic acid (*XXII*) from *XVI*.

2-R-3-R^1-4-R^2-Furo[3,2-*b*]pyrrole-5-carbohydrazides (*XXIII* – *XXIX*)

Hydrazine hydrate (88%, 3.5 g) was added to a solution of corresponding esters (10 mmol) in ethanol (60 ml). The mixture was refluxed for 20 h, cooled and separated precipitate was filtered off. The compounds *XXIII* – *XXIX* were crystallized from ethanol. Analogously was prepared 1-benzylbenzo[*b*]furo[3,2-*b*]pyrrole-2-carbohydrazide (*XXX*).

1-Benzylbenzo[*b*]furo[3,2-*b*]pyrrole (*XXXI*)

A solution of 1-acetylbenzo[*b*]furo[3,2-*b*]pyrrole (1.99 g, 0.01 mol) in ethanol (25 ml) was refluxed with sodium hydroxide (5%, 15 ml) for 20 min and poured on ice. Extraction with ether, washing the extract with 0.1 M hydrochloric acid and water drying over sodium sulfate and evaporation of solvent afforded *XXXI*.

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