AMIDES OF 5-NITRO-3-FURANECARBOXYLIC ACID

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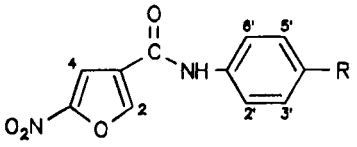
β-Substituted furane derivatives such as N-(4-R-phenyl)-5-nitro-3-furanecarboxamides (*Ia – Ij*) have been prepared by the reaction of 5-nitro-3-furanoyl chloride with 4-R-anilines (R = H, CH_3, OCH_3, $N(CH_3)_2$, Cl, Br, $NHCOCH_3$, CN, $COOCH_3$, NO_2). The structure of the compounds synthesized has been confirmed by their IR, UV, ^1H NMR and mass spectra.



Ia – Ij

EXPERIMENTAL

The melting points were measured with a Kofler apparatus. The IR spectra were recorded with a Perkin–Elmer 577 spectrophotometer, the UV spectra (λ in nm, ε in $m^2\ mol^{-1}$) were measured with a Specord UV VIS apparatus (Zeiss, Jena). The ^1H NMR spectra were measured in hexadeuteriodimethyl sulfoxide solutions using a Tesla BS 487C apparatus (80 MHz) with tetramethylsilane as the internal standard. The mass spectra (EI) were measured with an AEI (Manchester) MS 902 S spectrometer (direct inlet system), ionization energy 70 eV, trap current 100 μA, temperature of ion source 150 °C.

N-(4-R-Phenyl)-5-nitro-3-furanecarboxamides (*Ia – Ij*)

A solution of 8 mmol 4-R-aniline in 10 ml dioxane was stirred at 10 – 15 °C, and a solution of 5-nitro-3-furanoyl chloride[1] (0.6 g, 4 mmol) in 10 ml dioxane was added. The stirring was continued for another 3 h, whereafter the reaction mixture was poured into the same volume of water and acidified with dilute hydrochloric acid to a slightly acid reaction. The solid formed was collected by filtration and washed with water and with ether. After drying the product was recrystallized from a suitable solvent. Tables I – III give characteristic data for the coumpounds *Ia – Ij*.

TABLE I
Physico-chemical data of N-(4-R-phenyl)-5-nitro-3-furanecarboxamides (*Ia – Ij*)

Compound R	Formula (M. w.)	M. p., °C (Yield, %)	Calculated/Found			Mass spectrum M^+, *m/z*
			% C	% H	% N	
Ia	$C_{11}H_8N_2O_4$	164 – 166[a]	56.90	3.47	12.06	232
H	(232.2)	(99)	55.58	3.78	12.15	
Ib	$C_{12}H_{10}N_2O_4$	178 – 180[a]	58.54	4.09	11.38	246
CH_3	(246.2)	(89)	58.80	4.13	11.31	
Ic	$C_{12}H_{10}N_2O_5$	182 – 184[b, c]	54.97	3.84	10.68	262
OCH_3	(262.2)	(95)	54.81	3.61	10.51	
Id	$C_{13}H_{13}N_3O_4$	244 – 246[b]	56.73	4.76	15.27	275
$N(CH_3)_2$	(275.3)	(84)	56.90	4.65	15.47	
Ie	$C_{11}H_7ClN_2O_4$	202 – 204[a]	49.55	2.65	10.51	266/268
Cl	(226.6)	(99)	49.39	2.70	10.37	
If	$C_{11}H_7BrN_2O_4$	227 – 229[a]	42.47	2.27	9.00	310/312
Br	(311.1)	(94)	42.32	2.31	8.99	
Ig	$C_{13}H_{11}N_3O_5$	236 – 238[b]	53.98	3.83	14.53	289
$NHCOCH_3$	(289.2)	(97)	53.00	4.31	14.87	
Ih	$C_{12}H_7N_3O_4$	279 – 282[b]	56.04	2.74	16.34	257
CN	(257.2)	(85)	55.70	2.70	15.93	
Ii	$C_{13}H_{10}N_2O_6$	205 – 207[a]	53.80	3.47	9.65	290
$COOCH_3$	(290.2)	(95)	53.49	3.51	9.58	
Ij	$C_{11}H_7N_3O_6$	236 – 237[a, d]	47.66	2.55	15.16	277
NO_2	(277.2)	(98)	48.00	2.69	15.08	

Recrystallized from: [a] methanol; [b] ethanol; [c] ref.[2] gives m.p. 184 – 185 °C; [d] ref.[2] gives m.p. 236 – 237 °C.

TABLE II
IR (in KBr) and UV (in dioxane, c 5 . 10^{-5} mol l^{-1}) spectra of compounds *Ia – Ij*

Compound	IR spectrum, cm^{-1}		UV spectrum	
	ν(N–H)	ν(C=O)	λ_{max}, nm	log ε
Ia	3 260	1 645	283	3.11
Ib	3 400	1 675	291	3.16
Ic	3 410	1 670	297	3.16
Id	3 380	1 640	315	3.28
Ie	3 398	1 660	287	3.78
If	3 398	1 675	285	3.31
Ig	3 306	1 651	287	3.25
Ih	3 330	1 692	284	3.39
Ii	3 340	1 680	294	3.38
Ij	3 350	1 690	316	3.26

TABLE III
The ^1H NMR spectral parameters (δ, ppm) of N-(4-R-phenyl)-5-nitro-3-furanecarboxamides (*Ia – Ij*) in $(CD_3)_2SO$

Com-pound	H-2[a]	H-4	H-2′ H-6′	H-3′ H-5′	NH	Other signals
Ia	8.68 d	8.17 d	*b*	*b*	10.23 bs	*b*
Ib	8.66 d	8.16 d	7.60 m	7.18 m	10.15 bs	2.28 s (CH_3)
Ic	8.65 d	8.15 d	7.62 m	6.95 m	10.13 bs	3.75 s (OCH_3)
Id	8.62 d	8.14 d	7.51 m	6.73 m	9.98 bs	2.87 s (CH_3)
Ie	8.69 d	8.16 d	7.76 m	7.42 m	10.34 bs	–
If	8.69 d	8.15 d	7.70 m	7.55 m	10.32 bs	–
Ig	8.66 d	8.15 d	7.59 m	7.59 m	10.16 bs	2.04 s (CH_3)
Ih	8.74 d	8.17 d	7.85 m	7.91 m	10.59 bs	–
Ii	8.72 d	8.17 d	7.88 m	7.98 m	10.50 bs	3.84 s (OCH_3)
Ij	8.75 d	8.18 d	7.99 m	8.28 m	10.73 bs	–

[a] $J(2,4)$ = 1.1 Hz; [b] 6.80 – 7.80 m, 5 H (H-arom).

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