

REACTION OF ETHYL 5-SUBSTITUTED-2-FUROYLMALONATES WITH SECONDARY AMINES

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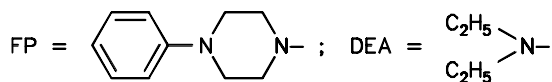
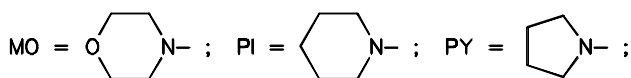
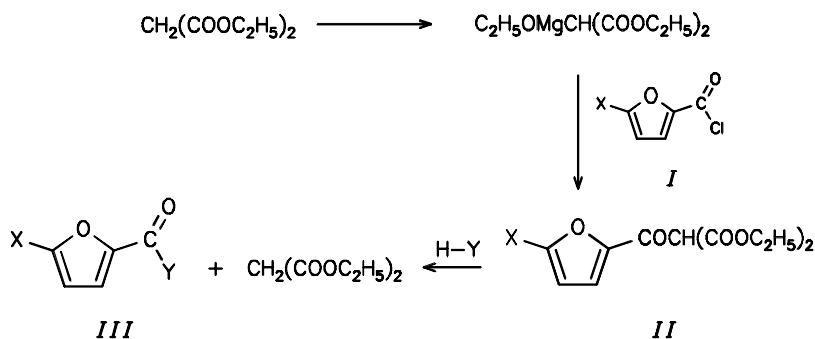
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In situ prepared magnesium salts of ethyl malonate react with 5-X-2-furoyl chlorides (X = Br, NO₂, C₆H₅S and C₆H₅SO₂) to give the corresponding ethyl 5-X-2-furoylmalonates. On treatment of these compounds with secondary amines no nucleophilic substitution of the group X in position 5 of the furan nucleus took place but, instead, amides of 5-X-2-furancarboxylic acids were isolated.

In our previous studies on reactions of furan derivatives with nucleophilic reagents we described nucleophilic substitution of the arylthio, heteroarylthio, arylsulfonyl and aryl-oxy groups with nitrogen nucleophilic reagents in 2-cyano-3-(5-arylthio-2-furyl)-, 2-cyano-3-(5-heteroarylthio-2-furyl)-, 2-cyano-3-(5-arylsulfonyl-2-furyl)- and 2-cyano-3-(5-aryloxy-2-furyl)acrylonitrile or -metacrylate¹⁻⁵. At the same time we have shown that the S_N reaction in position 5 of the furan nucleus takes place thanks to the presence of a strong electron-accepting substituent in position 2. This finding is confirmed also by studies of condensations of 5-X-furaldehydes with ethyl malonate, ethyl acetoacetate and acetylacetone with secondary amines which afforded products of S_N and Ad_N reactions⁶.

The present study concerns the preparation of ethyl 5-X-2-furoylmalonates (X = Br, NO₂, C₆H₅S, C₆H₅SO₂) and their reactions with secondary amines. The ethyl 5-X-2-furoylmalonates were prepared using a method described by Bowman⁷, which is based on reaction of ethoxymagnesium salt of ethyl malonate (prepared in situ) with 5-X-2-furoyl chloride in an inert solvent and which leads unequivocally to C-acylated products (Scheme 1). The reaction time was 2 h in the case of compounds *Iia*, *Iic*, *Iid* but only 1 h for derivative *Iib*. In the latter case, prolongation of the reaction time resulted in darkening of the reaction mixture, formation of tars and drop in the yield. The starting 5-X-2-furoyl chlorides *Ia* – *Id* were prepared by reaction of the corresponding 5-X-2-furancarboxylic acids with thionyl chloride. When the thionyl chloride was added to the carboxylic acid in one portion and the reaction mixture was refluxed, the reaction was accompanied by formation of black tar and the yield decreased to 70%. On the



<i>I, II</i>	X	<i>I, II</i>	X
<i>a</i>	Br	<i>c</i>	C ₆ H ₅ S
<i>b</i>	NO ₂	<i>d</i>	C ₆ H ₅ SO ₂

<i>III</i>	X	Y	<i>III</i>	X	Y	<i>III</i>	X	Y	<i>III</i>	X	Y
<i>a</i>	Br	MO	<i>f</i>	NO ₂	MO	<i>k</i>	C ₆ H ₅ S	MO	<i>p</i>	C ₆ H ₅ SO ₂	MO
<i>b</i>	Br	PI	<i>g</i>	NO ₂	PI	<i>l</i>	C ₆ H ₅ S	PI	<i>r</i>	C ₆ H ₅ SO ₂	PI
<i>c</i>	Br	PY	<i>h</i>	NO ₂	PY	<i>m</i>	C ₆ H ₅ S	PY	<i>s</i>	C ₆ H ₅ SO ₂	PY
<i>d</i>	Br	FP	<i>i</i>	NO ₂	FP	<i>n</i>	C ₆ H ₅ S	FP	<i>t</i>	C ₆ H ₅ SO ₂	FP
<i>e</i>	Br	DEA	<i>j</i>	NO ₂	DEA	<i>o</i>	C ₆ H ₅ S	DEA	<i>u</i>	C ₆ H ₅ SO ₂	DEA

SCHEME 1

other hand, when the reaction was carried out in anhydrous benzene and thionyl chloride was added during the reflux, the yield increased to 81 – 89%.

Ethyl 5-X-2-furoylmalonates have five potential sites suitable for a nucleophilic attack: the two ester groups, the carbonyl in position 2 of the furan ring, the position 5 of the furan ring and the strongly acidic hydrogen atom on the sp^3 -hybridized carbon atom. The more interesting is the fact that reactions with secondary amines afford solely amides of 5-X-2-furancarboxylic acids under simultaneous expulsion of ethyl malonate which was detected by thin-layer chromatography. The reaction probably starts by nucleophilic addition to the keto group, followed by elimination of ethyl malonate molecule. Of 5-bromo- and 5-nitro-2-furancarboxylic acid amides, compounds *IIIe* (ref.⁹), *IIIj* (ref.^{8,9}), *IIIf* (ref.^{8,10,11}), *IIIg* (ref.⁸) and *IIIh* (ref.¹¹) are already described: they were prepared from the corresponding acid chlorides and secondary amines.

EXPERIMENTAL

Infrared absorption spectra (ν , cm^{-1}) were measured on a Specord M 80 (Zeiss, Jena) instrument in KBr pellets (unless stated otherwise), UV spectra (in methanol) were obtained with a Specord M 40, (Zeiss, Jena) spectrometer. ^1H NMR spectra (δ , ppm; J , Hz) were measured on a BS 487 C Tesla instrument (80 MHz) in deuteriochloroform (compounds *I* and *II*) and hexadeuterioacetone (compounds *III*) with tetramethylsilane as internal standard.

The 5-bromo-, 5-phenylthio- and 5-phenylsulfonyl-2-furancarboxylic acids were prepared by oxidation of the corresponding aldehydes with silver oxide¹². 5-Nitro-2-furancarboxylic acid was obtained starting from 2-furancarboxylic acid: its esterification and subsequent nitration afforded ethyl 5-nitro-2-furancarboxylate which upon acid hydrolysis gave 5-nitro-2-furancarboxylic acid in 94% yield¹³.

5-X-2-Furoyl Chlorides *I* (General Procedure)

Thionyl chloride (6.53 g, 0.055 mol) in anhydrous benzene (5 ml) was added in the course of 9 – 12 h to a refluxing solution of the appropriate 5-X-2-furancarboxylic acid (0.05 mol) and several drops of distilled dimethylformamide in anhydrous benzene (15 ml). After removal of the benzene and excess thionyl chloride, the product was isolated by distillation under diminished pressure (compounds *Ia* – *Ic*). 5-Phenylsulfonyl-2-furoyl chloride (*Id*) separated from the reaction mixture as a white crystalline compound which was collected, washed with anhydrous benzene (2 – 3 times 10 ml) and dried.

5-Bromo-2-furoyl chloride (*Ia*). Yield 87%, b.p. 78 – 80 °C/2.39 kPa (reported¹⁴ b.p. 89 °C/107 kPa). IR spectrum (chloroform): 1 735 (CO). UV spectrum (λ_{max} , nm (log ϵ)): 215 (2.53), 268 (3.00), 284 sh (2.98). ^1H NMR spectrum: 7.63 d, 1 H, $J(3,4) = 3.75$ (H-3); 6.86 d, 1 H (H-4).

5-Nitro-2-furoyl chloride (*Ib*). Yield 82%, b.p. 129 – 131 °C/1.59 kPa (reported¹⁵ b.p. 110 – 123 °C/0.53 kPa). IR spectrum (chloroform): 1 760 (CO). UV spectrum (λ_{max} , nm (log ϵ)): 212 (3.22), 285 (3.33). ^1H NMR spectrum: 7.73 d, 1 H, $J(3,4) = 4.00$ (H-3); 7.92 d, 1 H (H-4).

5-Phenylthio-2-furoyl chloride (*Ic*). Yield 89%, b.p. 140 – 142 °C/0.16 kPa. For $\text{C}_{12}\text{H}_7\text{ClO}_2\text{S}$ (238.7) calculated: 55.35% C, 2.96% H, 13.43% S; found: 55.23% C, 2.95% H, 13.52% S. IR spectrum (chloroform): 1 754 (CO). UV spectrum (λ_{max} , nm (log ϵ)): 242 i (3.15), 252 (3.19), 292 (2.68). ^1H NMR spectrum: 7.67 d, 1 H, $J(3,4) = 3.80$ (H-3); 6.79 d, 1 H (H-4); 7.33 – 7.45 m, 5 H (H-aryl).

5-Phenylsulfonyl-2-furoyl chloride (Id). Yield 83%, m.p. 112 – 113 °C. For $C_{11}H_7ClO_4S$ (270.7) calculated: 48.78% C, 2.60% H, 11.84% S; found: 48.69% C, 2.78% H, 11.76% S. IR spectrum (chloroform): 1 760 (CO). UV spectrum (λ_{max} , nm (log ϵ)): 222 (3.04), 261 (3.31), 269 i (3.19). 1H NMR spectrum: 7.59 d, 1 H, $J(3,4) = 3.80$ (H-3); 7.43 d, 1 H (H-4); 7.70 – 7.78 m, 3 H (H-3', H-5'); 8.00 – 8.33 m, 2 H (H-2', H-6').

Ethyl 5-X-2-Furoylmalonates II (General Procedure)

Approximately 2 ml of a solution of ethyl malonate (16 g, 0.1 mol) in anhydrous ethanol (4.6 ml) and anhydrous benzene (20 ml) was added dropwise to a stirred mixture of magnesium (2.4 g, 0.1 mol), anhydrous benzene (7.33 ml), anhydrous ethanol (0.6 ml) and carbon tetrachloride (0.5 ml). The mixture was warmed until a vigorous reaction spontaneously commenced. The remaining solution of the malonate was gradually added so as to keep the mixture boiling (about 2 – 3 h). The mixture was then heated until all the magnesium reacted and the alcohol and benzene were removed by distillation. A solution of the corresponding furoyl chloride *I* (0.05 mol) in benzene was added dropwise to the residue under cooling and the mixture was refluxed for 1 – 2 h. After cooling, the mixture was decomposed with ice and dilute sulfuric acid, the organic layer was separated, washed with dilute sulfuric acid, several times with water, and dried over sodium sulfate. The benzene and excess ethyl malonate were distilled off and the crude product was purified by distillation in vacuo.

Ethyl 5-bromo-2-furoylmalonate (IIa). Yield 83%, b.p. 138 – 140 °C/2 kPa. For $C_{12}H_{13}BrO_6$ (333.1) calculated: 43.26% C, 3.93% H; found 43.21% C, 3.91% H. IR spectrum (chloroform): 1 750 (CO ester), 1 690 (CO ketone). UV spectrum (λ_{max} , nm (log ϵ)): 202 (2.78), 217 (2.56), 290 (3.26). 1H NMR spectrum: 1.20 t, 6 H, $J = 7.0$ (CH_3); 4.20 q, 4 H, $J = 7.0$ (CH_2); 5.25 s, 1 H (CH); 7.42 d, 1 H, $J(3,4) = 3.50$ (H-3); 6.75 d, 1 H (H-4).

Ethyl 5-nitro-2-furoylmalonate (IIb). Yield 77%, b.p. 155 – 159 °C/0.12 kPa. For $C_{12}H_{13}NO_8$ (299.2) calculated: 48.17% C, 4.38% H, 4.67% N; found: 48.09% C, 4.41% H, 4.52% N. IR spectrum: 1 740 (CO ester), 1 650 (CO ketone). UV spectrum (λ_{max} , nm (log ϵ)): 225 (2.93), 285 sh (2.98), 309 (3.06). 1H NMR spectrum: 1.23 t, 6 H, $J = 7.0$ (CH_3); 4.25 q, 4 H, $J = 7.0$ (CH_2); 5.43 s, 1 H (CH); 7.65 s, 1 H (H-3); 7.65 s, 1 H (H-4).

Ethyl 5-phenylthio-2-furoylmalonate (IIc). Yield 66%, m.p. 59 – 60 °C. For $C_{18}H_{18}O_6S$ (362.4) calculated: 59.66% C, 5.01% H, 8.84% S; found: 59.42% C, 4.92% H, 8.68% S. IR spectrum (chloroform): 1 750 (CO ester), 1 690 (CO ketone). UV spectrum (λ_{max} , nm (log ϵ)): 240 (2.98), 278 (2.93), 322 (3.04). 1H NMR spectrum: 1.13 t, 6 H, $J = 7.0$ (CH_3); 4.13 q, 4 H, $J = 7.0$ (CH_2); 5.20 s, 1 H (CH); 7.45 d, 1 H, $J(3,4) = 3.5$ (H-3); 6.80 d, 1 H (H-4); 7.30 s, 5 H (H-aryl).

Ethyl 5-phenylsulfonyl-2-furoylmalonate (IId). Yield 75%, m.p. 25 – 27 °C. For $C_{18}H_{18}O_8S$ (394.4) calculated: 54.82% C, 4.60% H, 8.13% S; found: 54.51% C, 4.72% H, 8.02% S. IR spectrum (chloroform): 1 735 (CO ester), 1 700 (CO ketone). UV spectrum (λ_{max} , nm (log ϵ)): 221 (3.29), 261 (3.47), 266 i (3.40). 1H NMR spectrum: 1.15 t, 6 H, $J = 7.0$ (CH_3); 4.15 q, 4 H, $J = 7.0$ (CH_2); 5.33 s, 1 H (CH); 7.49 s, 1 H (H-3); 7.49 s, 1 H (H-4); 7.65 – 7.75 m, 3 H (H-3', H-4', H-5'); 7.95 – 8.13 m, 2 H (H-2', H-6').

5-X-2-Furancarboxylic Acid Amides III (General Procedure)

The appropriate secondary amine (0.05 mol) was added to a stirred solution of the ethyl 5-X-2-furoylmalonate (0.05 mol) in acetonitrile (10 ml) and the reaction mixture was stirred for 8 h. The reaction course was monitored by thin-layer chromatography. After the reaction had ended, the reaction mixture was cooled, the crude product collected on filter and crystallized from ethanol. The obtained compounds and their physicochemical properties are listed in Table I, their 1H NMR spectral data are given in Table II.

TABLE I
Physicochemical characteristics of 5-X-2-furancarboxylic acid amides

Compound	Formula M.w.	M.p., °C Yield, %	Calculated/Found				λ_{max} , nm (log ϵ)	$\nu(\text{C}=\text{O})$, cm^{-1}		
			% C	% H	% N	% S				
<i>IIIa</i>	C ₉ H ₁₀ BrNO ₃ 260.1	156 – 157 74	41.57 41.38	3.87 3.81	5.38 5.27	–	205 (2.71)	261 (3.11)	300 (2.08)	1 640
<i>IIIb</i>	C ₁₀ H ₁₂ BrNO ₂ 258.2	143 – 136 76	46.52 46.40	4.68 4.59	5.42 5.37	–	202 (2.34)	257 (2.62)	–	1 630
<i>IIIc</i>	C ₉ H ₁₀ BrNO ₂ 244.2	102 – 104 73	44.26 44.14	4.12 4.08	5.73 5.68	–	208 (2.65)	264 (3.22)	–	1 630
<i>IIId</i>	C ₁₅ H ₁₃ BrN ₂ O ₂ 335.3	113 – 115 79	53.72 53.56	4.51 4.43	8.35 8.18	–	202 (3.44)	252 (3.37)	–	1 620
<i>IIIe</i>	C ₉ H ₁₂ BrNO ₂ 246.2	130 ^e 65	–	–	–	–	209 (2.92)	274 (2.89)	311 sh (2.72)	1 610
<i>IIIf</i>	C ₉ H ₁₀ N ₂ O ₅ 226.2	115 – 117 ^b 73	–	–	–	–	215 (3.30)	257 (3.42)	306 (2.54)	1 640
<i>IIIg</i>	C ₁₀ H ₁₀ N ₂ O ₄ 224.2	93 – 95 ^b 70	–	–	–	–	216 (2.80)	284 (2.83)	307 (2.74)	1 635
<i>IIIh</i>	C ₉ H ₁₀ N ₂ O ₄ 210.2	158 – 159 ^b 78	–	–	–	–	217 (3.20)	–	308 (3.18)	1 630
<i>IIIi</i>	C ₁₅ H ₁₅ N ₃ O ₄ 301.3	134 – 136 75	59.79 59.58	5.01 4.88	13.94 13.80	–	203 (3.52)	247 (3.25)	303 (3.05)	1 640
<i>IIIj</i>	C ₉ H ₁₂ N ₂ O ₄ 212.2	158 – 160 ^f 77	–	–	–	–	214 (2.64)	259 (3.26)	–	1 680

TABLE I
(Continued)

Compound	Formula M.w.	M.p., °C Yield, %	Calculated/Found				λ_{max} , nm (log ϵ)	$\nu(\text{C}=\text{O})$, cm^{-1}
			% C	% H	% N	% S		
<i>IIIk</i>	$\text{C}_{15}\text{H}_{15}\text{NO}_3\text{S}$ 289.4	71 – 72 69	62.22 62.20	5.22 5.35	4.84 4.90	11.08 10.92	241 (3.12)	– 1 625
<i>IIIl</i>	$\text{C}_{16}\text{H}_{17}\text{NO}_2\text{S}$ 287.4	44 – 46 68	66.87 66.70	5.96 5.90	4.87 4.77	11.15 11.02	241 (3.29)	– 1 620
<i>IIIm</i>	$\text{C}_{15}\text{H}_{15}\text{NO}_2\text{S}$ 273.4	104 – 105 64	65.90 65.90	5.53 5.48	5.12 5.16	11.73 11.58	242 (3.19)	– 1 625
<i>IIIn</i>	$\text{C}_{21}\text{H}_{20}\text{N}_2\text{O}_2\text{S}$ 364.5	83 – 84 47	69.21 69.27	5.53 5.60	7.68 7.67	8.79 8.62	247 (3.44)	– 1 625
<i>IIIo</i>	$\text{C}_{15}\text{H}_{17}\text{NO}_2\text{S}$ 275.4	121 – 122 66	65.43 65.40	6.20 6.35	5.08 5.19	11.64 11.50	252 (3.22)	– 1 630
<i>IIIp</i>	$\text{C}_{15}\text{H}_{15}\text{NO}_5\text{S}$ 321.4	120 – 121 65	56.06 56.11	4.70 4.68	4.36 4.60	9.97 9.82	219 (3.08)	– 1 640
<i>IIIr</i>	$\text{C}_{16}\text{H}_{17}\text{NO}_4\text{S}$ 319.4	85 – 86 55	60.17 60.28	5.36 5.01	4.39 4.30	10.04 9.88	219 (3.13)	– 1 630
<i>IIIs</i>	$\text{C}_{15}\text{H}_{15}\text{NO}_4\text{S}$ 305.4	131 – 132 51	59.00 58.98	4.95 4.99	4.59 4.70	10.50 10.41	221 (3.11)	– 1 630
<i>IIIt</i>	$\text{C}_{21}\text{H}_{20}\text{N}_2\text{O}_4\text{S}$ 396.5	164 – 165 52	63.62 63.65	5.10 4.96	7.07 7.27	8.08 7.91	222 i (3.24)	– 1 650
<i>IIIv</i>	$\text{C}_{15}\text{H}_{17}\text{NO}_4\text{S}$ 307.4	136 – 137 67	58.62 58.69	5.57 5.61	4.56 4.60	10.43 10.29	218 (2.95)	– 1 640

^a Ref.⁹ b.p. 150 °C/6.66 kPa. ^b Ref.¹¹ for *IIIj* m.p. 115 – 116 °C, for *IIIg* m.p. 92 – 94 °C, for *IIIh* m.p. 159 – 161 °C. ^c Ref.⁹ b.p. 123 – 124 °C/0.053 kPa. i inflex, sh shoulder.

TABLE II
¹H NMR chemical shifts^a (δ, ppm) for 5-X-2-furancarboxylic acid amides III

Compound	H-3	H-4	X, Y
<i>IIIa</i>	6.98 d	6.63 d	3.68 s, 8 H (H-2, H-3)
<i>IIIb</i>	6.87 d	6.56 d	1.58 – 1.65 m, 6 H (H-3, H-4, H-5); 3.58 – 3.68 m, 4 H (H-2, H-6)
<i>IIIc</i>	7.00 d	6.60 d	3.50 t, 4 H (H-3, H-4); 3.77 t, 4 H (H-2, H-5)
<i>III d</i>	7.04 d	6.67 d	3.27 t, 4 H (H-3, H-5); 3.92 t, 4 H (H-4, H-6); 6.78 – 7.35 m, 5 H (H-aryl)
<i>IIIe</i>	6.85 d	6.52 d	1.12 t, 6 H (2 × CH ₃); 2.85 q, 4 H (2 × NCH ₂)
<i>III f</i>	7.18 d	7.54 d	3.70 s, 8 H (H-2, H-3)
<i>III g</i>	7.10 d	7.53 d	1.64 – 1.69 m, 6 H (H-3, H-4, H-5); 3.61 – 3.72 m, 4 H (H-2, H-6)
<i>III h</i>	7.25 d	7.56 d	3.56 t, 4 H (H-3, H-4); 3.89 t, 4 H (H-2, H-5)
<i>III i</i>	7.26 d	7.62 d	3.31 t, 4 H (H-3, H-5); 3.87 t, 4 H (H-2, H-6); 6.78 – 7.21 m, 5 H (H-aryl)
<i>III j</i>	7.05 d	7.50 d	1.16 t, 6 H (2 × CH ₃); 3.06 q, 4 H (2 × NCH ₂)
<i>III k</i>	7.00 d	6.85 d	7.26 s, 5 H (H-aryl); 3.61 s, 8 H (H-2, H-3)
<i>III l</i>	7.10 d	6.90 d	7.29 s, 5 H (H-aryl); 1.58 – 1.65 m, 6 H (H-3, H-4, H-5); 3.42 – 3.65 m, 4 H (H-2, H-6)
<i>III m</i>	7.11 d	6.91 d	7.31 s, 5 H (H-aryl); 3.53 t, 4 H (H-3, H-4); 3.75 t, 4 H (H-2, H-6)
<i>III n</i>	7.09 d	6.94 d	7.36 s, 5 H (H-aryl); 3.21 t, 4 H (H-3, H-5); 3.86 t, 4 H (H-2, H-6); 7.00 – 7.28 m, 5 H (H-aryl)
<i>III o</i>	6.92 d	6.86 d	7.27 s, 5 H (H-aryl); 1.11 t, 6 H (2 × CH ₃); 2.80 q, 4 H (2 × NCH ₂)
<i>III p</i>	7.38 d	7.06 d	7.66 – 7.80 m, 3 H (<i>m, p</i>); 7.98 – 8.12 m, 2 H (<i>o</i>); 3.64 s, 8 H (H-2, H-3)
<i>III r</i>	7.39 d	7.00 d	7.65 – 7.80 m, 3 H (<i>m, p</i>); 7.99 – 8.13 m, 2 H (<i>o</i>); 1.55 – 1.68 m, 6 H (H-3, H-4, H-5); 3.49 – 3.59 m, 4 H (H-2, H-6)
<i>III s</i>	7.39 d	7.14 d	7.66 – 7.82 m, 3 H (<i>m, p</i>); 8.00 – 8.15 m, 2 H (<i>o</i>); 3.51 t, 4 H (H-3, H-4); 3.75 t, 4 H (H-2, H-5)
<i>III t</i>	7.42 d	7.12 d	7.78 – 7.80 m, 3 H (<i>m, p</i>); 8.03 – 8.14 m, 2 H (<i>o</i>); 3.28 t, 4 H (H-3); 3.84 t, 4 H (H-2); 6.85 – 7.36 m, 5 H (H-aryl)
<i>III v</i>	7.30 d	6.96 d	7.57 – 7.72 m, 3 H (<i>m, p</i>); 7.00 – 8.05 m, 2 H (<i>o</i>); 1.15 t, 6 H (2 × CH ₃); 3.00 q, 4 H (2 × NCH ₂)

^a *J*(3,4) = 3.5 – 3.8 Hz.

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