

N-Substituted 2,6-dialkylanilides

^aA. KRUTOŠÍKOVÁ, ^aM. DANDÁROVÁ, ^bV. KONEČNÝ, and ^aJ. KOVÁČ

^aDepartment of Organic Chemistry, Faculty of Chemical Technology,
Slovak Technical University, CS-81237 Bratislava

^bResearch Institute of Chemical Technology, CS-83106 Bratislava

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Dedicated to Professor P. Kristian, DrSc., in honour of his 60th birthday

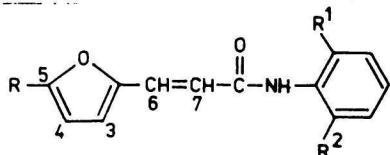
By reactions of 2,6-dialkylanilines or *N*-substituted 2,6-dialkylanilines with 3-(2-furyl)propenoyl chloride, 3-(5-nitro-2-furyl)propenoyl chloride and with other acyl chlorides the new 2,6-dialkylanilides or their *N*-substituted derivatives were prepared and their fungicidal and herbicidal properties were studied.

С помощью реакции 2,6-диалкиланилинов или *N*-замещенных 2,6-диалкиланилинов с 3-(2-фурил)пропеноилхлоридом, 3-(5-нитро-2-фурил)пропеноилхлоридом и другими ацилхлоридами были получены новые 2,6-диалкиланилиды или их *N*-замещенные производные, а также исследованы их фунгицидные и гербицидные свойства.

Many *N*-substituted 2,6-dialkylanilides are biologically active compounds [1—4]. One of them, 2-ethyl-6-methyl-*N*-(1-methoxy-2-propyl)chloroacetanilide (Metolachlor), is an active part of the antigrass herbicide Dual [1]. Metolachlor is a mixture of four stereoisomers, which were made and their absolute configuration was fixed by X-ray analysis [5].

The aim of this work was the synthesis of new type 2,6-disubstituted anilides and their *N*-substituted derivatives and the study of their fungicidal and herbicidal properties. The above-mentioned compounds were prepared by reactions of 2,6-dialkylanilines with 3-(2-furyl)propenoyl chloride or 3-(5-nitro-2-furyl)propenoyl chloride (compounds I—VI, Table 1). By treatment of the mentioned chlorides with *N*-(2-furyl)-2,6-dialkylanilines the compounds VII—XII (Table 2) were obtained. 2-Ethyl-6-methyl-*N*-[5-(3,4-dichlorophenyl)-2-furfuryl]aniline with the introduced chlorides, chloroacetyl as well as benzoyl chloride gave again the compounds XIII—XVI (Table 3). Analogously the above-mentioned chlorides with 2,6-dialkyl-*N*-(1-hydroxy or 1-methoxy)-2-propylanilines gave compounds XVII—XXIV (Table 4). The compounds XXV—XXIX were prepared by the reaction of 3-(2-furyl)propenoyl chloride and 3-(5-nitro-2-furyl)propenoyl chloride with 2,6-dialkyl-*N*-(1-methoxycarbonyl-1-ethyl)anilines (Table 5). The studied reactions were realized in benzene in the presence of water-free sodium carbonate (Scheme 1).

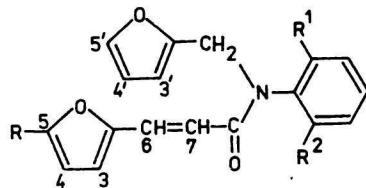
Table 1



Compound	R	R ¹	R ²	Formula	<i>M</i> _r	<i>w_i</i> (calc.)/%		Yield	M.p. °C	
						<i>w_i</i> (found)/%	C	H	N	%
<i>I</i>	CH ₃	CH ₃	CH ₃	C ₁₅ H ₁₅ NO ₂	241.3	74.66 75.01	6.26 6.21	5.80 5.98	78	184—187
<i>II</i>	H	CH ₃	C ₂ H ₅	C ₁₆ H ₁₇ NO ₂	255.3	75.27 75.39	6.71 6.60	5.49 5.78	82	201—202
<i>III</i>	H	C ₂ H ₅	C ₂ H ₅	C ₁₇ H ₁₉ NO ₂	269.3	75.82 75.42	7.11 7.18	5.20 5.27	93	187—190
<i>IV</i>	NO ₂	CH ₃	CH ₃	C ₁₅ H ₁₄ N ₂ O ₄	286.3	62.93 62.73	4.92 4.82	9.78 9.58	97	201—202
<i>V</i>	NO ₂	CH ₃	C ₂ H ₅	C ₁₆ H ₁₆ N ₂ O ₄	300.3	63.99 63.90	5.37 5.27	9.32 9.22	97	192—195
<i>VI</i>	NO ₂	C ₂ H ₅	C ₂ H ₅	C ₁₇ H ₁₈ N ₂ O ₄	314.3	64.97 64.77	5.77 5.67	9.13 9.10	76	190—193

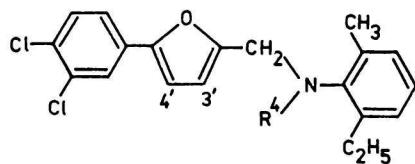
Table 2

2',6'-Dialkyl-N-(2-furyl)-3-(5-R-2-furyl)propenylanilides VII—XII



Compound	R	R ¹	R ²	Formula	M_r	$w_i(\text{calc.})/\%$			Yield %	M.p. °C
						C	H	N		
VII	H	CH ₃	CH ₃	C ₂₀ H ₁₉ NO ₃	321.4	74.74 74.26	5.96 5.86	4.35 4.32	80	94—95
VIII	H	CH ₃	C ₂ H ₅	C ₂₁ H ₂₁ NO ₃	335.4	75.20 74.94	6.31 6.18	4.18 4.56	90	100—102
IX	H	C ₂ H ₅	C ₂ H ₅	C ₂₂ H ₂₃ NO ₃	349.4	76.63 75.64	6.63 6.81	4.00 4.11	78	104—106
X	NO ₂	CH ₃	CH ₃	C ₂₀ H ₁₈ N ₂ O ₅	366.4	65.56 65.86	4.95 4.97	7.64 7.64	94	151—154
XI	NO ₂	CH ₃	C ₂ H ₅	C ₂₁ H ₂₀ N ₂ O ₅	380.4	66.31 66.39	5.30 5.27	7.36 7.30	96	92—96
XII	NO ₂	C ₂ H ₅	C ₂ H ₅	C ₂₂ H ₂₂ N ₂ O ₅	394.4	67.00 66.82	5.62 5.48	7.10 7.00	94	100—105

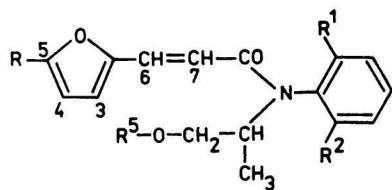
Table 3

2'-Ethyl-6'-methyl-N-[5-(3,4-dichlorophenyl)-2-furyl]-R⁴-anilides XIII—XVI

Compound	R ⁴	Formula	M _r	w _i (calc.)/%				Yield	M.p.
				C	H	Cl	N		
XIII	ClCH ₂ CO	C ₂₂ H ₂₀ Cl ₃ NO ₂	436.7	60.51 60.12	4.62 4.42	24.35 25.82	3.20 3.12	81	Yellow oil
XIV	vz. 2 do tab. 3	C ₂₇ H ₂₃ Cl ₂ NO ₂	464.4	69.83 69.63	4.99 4.79	15.27 15.07	3.07 3.18	76	122—123
XV		C ₂₇ H ₂₃ Cl ₂ NO ₃	480.4	67.50 67.36	4.83 4.75	14.75 14.62	2.91 2.73	74	133—135
XVI		C ₂₇ H ₂₂ Cl ₂ N ₂ O ₅	525.4	61.72 61.52	4.12 4.12	13.49 13.33	5.33 5.11	88	164—167

Table 4

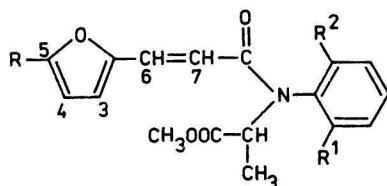
2',6'-Dialkyl-N-(1-hydroxy or 1-methoxy-2-propyl)-3-(2-furyl)- and -3-(5-nitro-2-furyl)propananilides XVII—XXIV



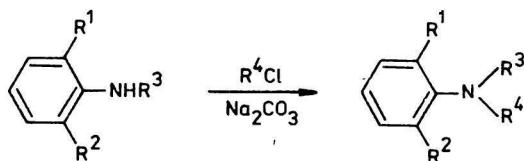
Compound	R	R ¹	R ²	R ⁵	Formula	M _r	<i>w_i</i> (calc.)/%			Yield	M.p.
							C	H	N		
							%	°C			
XVII	H	CH ₃	CH ₃	H	C ₁₈ H ₂₁ NO ₃	299.3	72.23 72.03	7.07 7.12	4.68 4.82	62	77—79
XVIII	H	CH ₃	C ₂ H ₅	H	C ₁₉ H ₂₃ NO ₃	313.4	72.82 72.88	7.40 7.21	4.59 4.02	72	82—83
XIX	NO ₂	CH ₃	CH ₃	H	C ₁₈ H ₂₀ N ₂ O ₅	340.3	63.53 63.56	5.92 5.90	8.23 7.88	76	125—126
XX	H	CH ₃	CH ₃	CH ₃	C ₁₉ H ₂₃ NO ₃	313.4	72.82 72.48	7.40 7.26	4.59 4.22	78	68—71
XXI	H	C ₂ H ₅	CH ₃	CH ₃	C ₂₀ H ₂₅ NO ₃	327.4	73.37 73.15	7.70 7.62	4.29 4.21	69	Yellow oil
XXII	NO ₂	CH ₃	CH ₃	CH ₃	C ₁₉ H ₂₂ N ₂ O ₅	358.4	63.67 63.82	6.19 6.10	7.82 7.78	96	126—128
XXIII	NO ₂	CH ₃	C ₂ H ₅	CH ₃	C ₂₀ H ₂₄ N ₂ O ₅	372.4	64.50 64.32	6.50 6.40	7.52 7.42	93	92—94
XXIV	NO ₂	C ₂ H ₅	C ₂ H ₅	CH ₃	C ₂₁ H ₂₆ N ₂ O ₅	386.4	65.28 65.08	6.78 6.60	7.25 7.05	93	77—82

Table 5

2',6'-Dialkyl-N-(1-methoxycarbonyl-1-ethyl)-3-(2-furyl or 5-nitro-2-furyl)propenylanilides XXV—XXIX



Compound	R	R ¹	R ²	Formula	<i>M</i> _r	<i>w_i</i> (calc.)/%			Yield	M.p.
						C	H	N		
XXV	H	CH ₃	CH ₃	C ₁₉ H ₂₁ NO ₄	327.4	69.70 69.20	6.46 6.06	4.27 4.68	76	Yellow oil
XXVI	H	CH ₃	C ₂ H ₅	C ₂₀ H ₂₃ NO ₄	341.4	70.36 70.06	6.79 7.02	4.10 4.04	72	Yellow oil
XXVII	NO ₂	CH ₃	CH ₃	C ₁₉ H ₂₀ N ₂ O ₆	372.4	61.28 61.36	5.41 5.48	7.52 7.70	95	174—175
XXVIII	NO ₂	CH ₃	C ₂ H ₅	C ₂₀ H ₂₂ N ₂ O ₆	386.4	62.17 62.60	5.74 5.54	7.25 7.17	84	119—122
XXIX	NO ₂	C ₂ H ₅	C ₂ H ₅	C ₂₁ H ₂₄ N ₂ O ₆	400.4	63.00 63.20	6.04 6.40	6.99 7.15	90	132—134



R^1, R^2 = methyl, ethyl

R^3 = H, 2-furfuryl, 5-(3,4-dichlorophenyl)-2-furfuryl, 1-hydroxy-2-propyl, 1-methoxy-2-propyl, 1-methoxycarbonyl-1-ethyl

R^4 = chloroacetyl, 3-(2-furyl)propenoyl, 3-(5-nitro-2-furyl)propanoyl, benzoyl

Scheme 1

The prepared compounds are yellow or orange coloured substances. Some of them, namely 2'-ethyl-6'-methyl-substituted products were relatively difficult to crystallize, which can be explained by the presence of four stereoisomers in the reaction products.

The structure of the synthesized compounds was proved by 1H NMR spectra (Tables 6—10). The data showed that the rotation around N -phenyl bond is broken down. 1H NMR data confirmed an atropisomerism in case the substituents are different.

Table 6

1H NMR data^a (δ /ppm; J /Hz) of the compounds I—VI

Proton $J_{H,H}$	Compound					
	I	II	III	IV	V	VI
H-3	6.75 dd	6.72 dd	6.70 dd	7.08 d	7.06 d	7.04 d
H-4	6.59 dd	6.54 dd	6.55 dd	7.58 d	7.57 d	7.56 d
H-5	7.69 dd	7.64 dd	7.69 dd	—	—	—
H-6	7.49 d	7.44 d	7.44 d	7.53 d	7.52 d	7.52 d
H-7	6.81 d	6.76 d	6.78 d	7.08 d	7.08 d	7.09 d
H_{arom}	7.05 bs	7.08 bs	7.12 bs	7.09 bs	7.18 bs	7.15 bs
R^1	2.22 s	2.18 s	2.60 q	2.22 s	2.22 s	2.62 q
			1.12 t			1.12 t
R^2	2.22 s	2.60 q	2.60 q	2.22 s	2.62 q	2.62 q
		1.10 t	1.12 t		1.12 t	1.12 t
NH	8.81 bs	8.72 bs	8.75 bs	9.00 bs	9.00 bs	9.00 bs
$J_{3,4}$	3.2	3.2	3.2	3.9	3.9	3.9
$J_{6,7}$	15.5	15.5	15.5	15.6	15.6	15.6

a) Measured in hexadeuteroacetone.

Table 7

¹H NMR data^a (δ /ppm; J /Hz) of the compounds VII—XII

Proton $J_{H,H}$	Compound					
	VII	VIII	IX	X	XI	XII
H-3	6.63 dd	6.61 dd	6.65 dd	7.02 d	7.00 d	7.00 d
H-4	6.47 dd	6.43 dd	6.45 dd	7.49 d	7.45 d	7.47 d
H-5	7.45 dd	7.41 dd	7.45 dd	—	—	—
H-6	7.49 d	7.50 d	7.48 d	7.56 d	7.54 d	7.55 d
H-7	6.00 d	6.01 d	6.01 d	6.28 d	6.28 d	6.30 d
H-3'	6.22 dd	6.20 dd	6.20 dd	6.23 dd	6.20 d	6.22 dd
H-4'	6.33 dd	6.30 dd	6.30 dd	6.34 dd	6.30 dd	6.32 dd
H-5'	7.40 dd	7.37 dd	7.40 dd	7.42 dd	7.44 dd	7.40 dd
H _{arom}	7.12 bs	b	b	7.19 bs	b	c
CH ₂	4.85 s	4.72 d ^d	4.85 s	4.87 s	4.81 d ^d	4.87 s
		4.82 d			4.96 d	
R ¹	1.93 s	1.90 s	2.32 q	1.95 s	1.95 s	2.32 q
			1.03 t			1.05 t
R ²	1.93 s	2.35 q	2.32 q	1.95 s	2.37 q	2.32 q
		1.03 t	1.03 t		1.10 t	1.05 t
$J_{3,4}$	3.2	3.2	3.2	3.9	3.9	3.9
$J_{6,7}$	15.2	15.2	15.2	15.4	15.5	15.5

a) Measured in hexadeuteroacetone; b) 7.0—7.4 m; c) 7.2—7.3 m; d) $^2J = 15.0$ Hz.

The value of the vicinal coupling constant $J_{H-6,H-7} = 15.5\text{--}15.6$ Hz for the derivatives I—VI (Table 6) and VII—XII (Table 7) confirmed *E*-configuration. The protons of CH₂ group at nitrogen atom of the compounds with the symmetrically substituted benzene ring (VI, IX, X, XII) display as a singlet, with unsymmetrically substituted benzene ring (VIII, XI) as two doublets of the AB spin system with a geminal coupling constant $^2J_{H-a,H-b} = 15.0$ Hz. The nonequivalent proton signals of the methylene group of the unsymmetrically substituted compounds XIII and XV (Table 8) display the doublets of doublets. The chemical shift values of methylene protons of the compounds XIV and XVI are very close and they were observed as broadened signals (Table 9).

The compounds XVII—XXIV (Table 9) are *E*-isomers. The unequivalent methylene protons near the asymmetrical carbon atom of 1-methoxy-2-propyl group display the doublets of doublets due to the mutual geminal interaction and the interaction with the methine proton. The methylene protons of the compounds XVIII, XIX form a multiplet due to further interaction with the hydroxyl group proton. In the case of the compounds XVIII, XXI, XXIII (Table 9) with two different 2,6-substituents on the benzene ring two singlets of the

Table 8

¹H NMR data (δ/ppm; J/Hz) of the compounds XIII—XVI^a

Proton <i>J</i> _{H,H}	Compound			
	XIII	XIV	XV ^b	XVI ^c
H-3'	6.45 d	6.47 d	6.40 d	6.73 d
H-4'	7.02 d	7.02 d	6.98 d	7.30 d
CH ₂ —N	4.93 d ^d 4.79 d	4.95 bs	4.92 d ^d 4.82 d	4.92 s
CH ₃	1.96 s	2.00 s	1.92 s	1.95 s
CH ₃ —CH ₂	2.37 q	2.45 q	2.35 q	2.35 q
CH ₃ —CH ₂	1.00 t	0.95 t	0.98 t	1.00 t
H-6	—	—	7.47 d	6.41 d
H-7	—	—	5.90 d	4.92 d
H-3	—	—	6.78 dd	7.38 dd
H-4	—	—	6.50 dd	7.92 dd
H-5	—	—	7.61 dd	—
CH ₂ Cl	3.82 s	—	—	—
<i>J</i> _{3',4'}	3.2	3.4	3.2	3.5
<i>J</i> _{6,7}	—	—	15.2	15.2

a) Measured in hexadeuteriodimethyl sulfoxide, H_{arom}: 7.10—7.80 m; b) R is 3-(2-furyl)-propenoyl; c) R is 3-(5-nitro-2-furyl)propenoyl, R is numbered as XV and XVI (see Table 7); d) ²J = 15.0 Hz.

Table 9

¹H NMR data^a (δ/ppm; J/Hz) of the compounds XVII—XXIV

Proton <i>J</i> _{H,H}	Compound				
	XVII	XVIII	XIX	XX	XXI
H-3	6.65 dd	6.64 dd	6.98 d	6.61 dd	6.61 dd
H-4	6.44 dd	6.45 dd	7.46 d	6.43 dd	6.43 dd
H-5	7.43 dd	7.46 dd	—	7.42 dd	7.43 dd
H-6	7.45 d	7.42 d	7.50 d	7.43 d	7.40 d
H-7	5.92 d	5.92 d	6.20 d	5.91 d	5.91 d
H _{arom}	7.17 bs	b	7.21 bs	7.20 bs	b
CH ₂ —CH	3.80 m	3.80 m	3.90 m	3.47 dd 3.73 dd	3.45 dd 3.76 dd
CH	4.10 m	4.08 m	4.10 m	4.25 m	4.25 m
OH	4.50 t	4.40 t	4.27 t	—	—

Table 9 (Continued)

Proton $J_{H,H}$	Compound				
	XVII	XVIII	XIX	XX	XXI
OCH ₃	—	—	—	3.20 s	3.22 s
CH ₃ —CH	1.17 d	1.19 d	1.18 d	1.15 d	1.15 d
R ¹	2.22 s	2.17	2.21	2.18 s	2.18
		2.32 s			2.15 s
R ²	2.16 s	2.63	2.26	2.22 s	2.58
		2.53 q			2.53 q
		1.24			1.16 t
J _{3,4}	3.3	3.3	3.8	3.3	3.3
J _{6,7}	15.1	15.1	15.1	15.2	15.2

Table 9 (Continued)

Proton $J_{H,H}$	Compound		
	XXII	XXIII	XXIV
H-3	6.98 d	6.97 d	6.97 d
H-4	7.48 d	7.46 d	7.47 d
H-6	7.49 d	7.47 d	7.47 d
H-7	6.20 d	6.22 d	6.24 d
H _{arom}	7.22 bs	b	7.32 bs
CH ₂ —CH	3.60 dd 3.72 dd	3.46 dd 3.74 dd	3.50 dd 3.75 dd
CH	4.25 m	4.25 m	4.25 m
OCH ₃	3.20 s	3.22 s	3.21 s
CH ₃ —CH	1.18 d	1.14 d	1.12 d
R ¹	2.20 s	2.20 s	2.56 q 1.18 t
R ²	2.22 s	2.75 q 1.20 t	2.61 q 1.18 t
J _{3,4}	4.0	4.0	4.0
J _{6,7}	15.5	15.5	15.5

a) Measured in hexadeuteroacetone; b) 7.10—7.30 m.

methyl group and two quartets of ethyl methylene group are observed due to atropisomerism. The methyl protons of the ethyl group were not distinguished. At the compounds XXVI and XXVIII having unsymmetrically substituted benzene ring the atropisomerism was observed as well (Table 10). The NMR

Table 10

¹H NMR data^a (δ /ppm; J/Hz) of the compounds XXV—XXIX

Proton $J_{H,H}$	Compound				
	XXV	XXVI	XXVII	XXVIII	XXIX
H-3	6.64 dd	6.65 dd	7.01 d	7.00 d	7.00 d
H-4	6.43 dd	6.45 dd	7.49 d	7.47 d	7.48 d
H-5	7.41 dd	7.46 dd	—	—	—
H-6	7.44 d	7.40 d	7.48 d	7.47 d	7.47 d
H-7	5.99 d	5.87 d	6.24 d	6.26 d	6.27 d
H _{arom}	7.22 bs	b	7.25 bs	b	b
<u>CH</u> —CH ₃	4.52 q 4.18 q	4.54 q	4.53 q	4.56 q 4.50 q	4.53 q
COOCH ₃	3.70 s	3.71 s	3.78 s	3.72 s	3.72 s
<u>CH</u> ₃ —CH	1.05 d 1.00 d	1.04 d	1.05 d	1.06 d 1.03 d	1.00 d
R ¹	2.42 s 2.12 s	2.41 s	2.41 s	2.41 s 2.13 s	2.50 q 1.17 t
R ²	2.15 s 1.15; 1.19	2.52; 2.91 1.15; 1.19	2.16 s	2.52; 2.92 1.17; 1.20	2.91 q 1.20 t
<i>J</i> _{3,4}	3.2	3.2	3.9	3.9	3.9
<i>J</i> _{6,7}	15.2	15.2	15.4	15.4	15.5

a) Measured in hexadeuteroacetone; b) 7.10—7.30 m.

spectra of these compounds display two quartets of methine proton and two doublets of the methyl group of 1-methoxycarbonyl-1-ethyl group attached at nitrogen atom.

Some of the prepared compounds exhibited the antifungal activity examined according to the standard method [6]. The compounds IV—VI, and XIX formed a sterile zone with tested *Phytoptora infestans*, the compounds IV—VI, XVIII, XXII—XXIV with *Botrytis cinerea* and the compounds IV—VII, XVIII, XXII—XXIV with *Fusarium niveale*.

None of the prepared compounds in standard test methods [7] on herbicidal activity reached the activity of the used standards.

Experimental

¹H NMR spectra were measured with Tesla BS 487 C apparatus operating at 80 MHz in hexadeuteroacetone and hexadeuterodimethyl sulfoxide at 25°C. Tetramethylsilane was used as internal reference. The obtained data and the solvents used are presented in Tables 6—10. The starting compounds were prepared according to [8—11].

2',6'-Dialkylanilides I—VI

To the suspension of 2,6-dialkylaniline (0.02 mol) and sodium carbonate (2.12 g; 0.02 mol) in benzene (10 cm³) 3-(2-furyl)- resp. 3-(5-nitro-2-furyl)propenoyl chloride (0.02 mol) in benzene (15 cm³) was added under stirring at 25 °C. The reaction mixture was stirred at room temperature for 1 h. The separated sodium chloride was filtered off, the solvent was distilled off and the residue was crystallized from ethanol.

The data of the compounds *I—VI* are given in Tables 1 and 6.

N-Substituted 2',6'-dialkylanilides VII—XXIX

To the suspension of *N*-substituted 2,6-dialkylaniline (0.02 mol) and sodium carbonate (2.12 g; 0.02 mol) in benzene (15 cm³) acyl chloride (0.02 mol) in benzene (15 cm³) was added under stirring at 25 °C (Scheme 1). The reaction mixture was stirred for 4 h, then filtered, the solvent was evaporated *in vacuo* and the residue was crystallized from ethanol.

In the case of the liquid products (*XIII, XXI, XXV, XXVI*) the reaction mixture after filtration was diluted with ethyl acetate, washed with water, 10 % hydrogen chloride and with saturated solution of sodium chloride. The organic layer was dried with sodium sulfate. The solvent was distilled off *in vacuo* and the residue was kept for 4 h *in vacuo* (*p* = 66 Pa) at 60 °C. The data of the compounds *VII—XXIX* are given in Tables 2—5, 7—10.

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