SYNTHESIS OF SOME TRIAZOLYL ACETANILIDES

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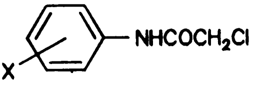
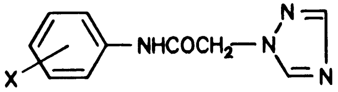
Derivatives of triazole constitute a group of compounds, interesting for their potential biological and phytoefectorical activity[1,2]. This has motivated us to prepare a series of substituted derivatives of triazolyl acetanilides (*IIa* – *IIn*) by reaction of 1-chloroacetanilides (*Ia* – *In*) with 1,2,4-triazole. The basic IR and ^1H NMR spectral data of final products are presented. Investigation of herbicidal, fungicidal and growth-regulation activity showed, that none of the studied compounds was more efficient that currently used compounds.



Ia – *In*



IIa – *IIn*

In formulae *I*, *II* :
a, X = H
b, X = 4-Cl
c, X = 3,4-Cl_2
d, X = 2,4-Cl_2
e, X = 3,5-Cl_2
f, X = 2,4,6-Cl_3
g, X = 2,3,4,5,6-Cl_5
h, X = 4-Br
i, X = 2,4-Br_2
j, X = 2,4,6-Br_3
k, X = 4-BrPh
l, X = 4-COPh
m, X = 4-F
n, X = 3-Br

EXPERIMENTAL

The IR spectra of compounds in KBr pellets were measured on Specord M 8 apparatus (Zeiss, Jena). The ^1H NMR spectra of hexadeuterioacetone solutions containing tetramethylsilane as an internal standard were recorded on Varian VXR-300 (300 MHz) spectrometer. Characteristic and spectral data are given in Tables I and II.

Table I
Characteristic data for prepared compounds

Compound[a]	Formula (M. w.)	M. p., °C Yield, %	Calculated/Found		
			% C	% H	% N
Ic	$C_8H_6Cl_3NO$	104 – 106	40.29	2.54	5.87
	(238.5)	89	40.11	2.49	5.79
Id	$C_8H_6Cl_3NO$	97 – 99	40.29	2.54	5.87
	(238.5)	88	40.10	2.48	5.78
Ie	$C_8H_6Cl_3NO$	137 – 139	40.29	2.54	5.87
	(238.5)	86	40.13	2.48	5.79
If	$C_8H_5Cl_4NO$	179 – 181	35.21	1.85	5.13
	(272.9)	80	35.19	1.87	5.08
Ig	$C_8H_3Cl_6NO$	207 – 209	28.11	0.88	4.10
	(341.8)	75	28.05	0.86	4.00
Ii	$C_8H_6Br_2ClNO$	107 – 109	29.35	1.85	4.28
	(327.4)	92	29.28	1.83	4.20
Ij	$C_8H_5Br_3ClNO$	202 – 208	23.65	1.24	3.44
	(406.3)	83	23.57	1.22	3.41
Ik	$C_{14}H_{11}BrClNO$	197 – 202	51.80	3.42	4.31
	(324.6)	82	51.70	3.38	4.29
Il	$C_{15}H_{12}ClNO_2$	133 – 134	65.83	4.42	5.12
	(273.7)	86	65.79	4.39	5.07
Im	C_8H_7ClFNO	129 – 131	51.22	3.76	7.46
	(187.6)	85	51.03	3.70	7.38
IIa	$C_{10}H_{10}N_4O$	135 – 136	59.39	4.98	27.70
	(202.2)	87	59.18	4.95	27.56
IIb	$C_{10}H_9ClN_4O$	199 – 202	50.73	3.83	23.67
	(236.7)	85	50.65	3.80	23.49
IIc	$C_{10}H_8Cl_2N_4O$	205 – 208	44.30	2.97	20.66
	(271.1)	79	44.21	2.91	20.50
IId	$C_{10}H_8Cl_2N_4O$	176 – 178	44.30	2.97	20.66
	(271.4)	81	44.71	2.90	20.49
IIe	$C_{10}H_8Cl_2N_4O$	177 – 179	44.30	2.97	20.66
	(271.4)	77	44.21	2.93	20.51
IIf	$C_{10}H_7Cl_3N_4O$	198 – 203	39.30	2.31	18.33
	(305.6)	73	39.22	2.20	18.25

TABLE I
(*Continued*)

Compound[a]	Formula (M. w.)	M. p., °C Yield, %	Calculated/Found		
			% C	% H	% N
IIg	$C_{10}H_5Cl_5N_4O$	230 – 231	32.08	1.35	14.96
	(374.4)	65	32.08	1.30	14.83
IIh	$C_{10}H_9BrN_4O$	196 – 198	42.73	3.23	19.93
	(281.1)	81	42.67	3.19	19.90
IIi	$C_{10}H_8Br_2N_4O$	191 – 193	33.36	2.24	15.56
	(360.0)	78	33.27	2.21	15.43
IIj	$C_{10}H_7Br_3N_4O$	256 – 260	27.36	1.60	14.76
	(438.9)	69	27.21	1.60	14.67
IIk	$C_{16}H_{13}BrN_4O$	276 – 280	53.80	3.67	15.68
	(357.2)	65	53.67	3.62	15.58
IIl	$C_{17}H_{14}N_4O_2$	193 – 196	66.66	4.61	18.29
	(306.3)	66	66.51	4.57	18.08
IIm	$C_{10}H_9FN_4O$	211 – 213	54.54	4.12	25.44
	(220.2)	70	54.39	4.07	25.37
IIn	$C_{10}H_9BrN_4O$	164 – 168	42.73	3.23	19.93
	(281.1)	75	42.60	3.19	19.89

[a] The compounds *Ia* (ref.[3]), *Ib* (ref.[5]), *Ih* (ref.[5]), *In* (ref.[4]) were already described.

1-Chloroacetanilides *Ia* – *Ie*, *Ih*, *Ik* – *In*

To the mixture of 0.01 mol substituted aniline dissolved in 15 ml benzene, 5 ml of 2 M aqueous solution of sodium hydroxide and 0.01 mol (1.2 g) chloroacetyl chloride in 5 ml benzene was added dropwise under stirring. The reaction mixture was stirred for 1 h. The separated product was filtered off, washed with water and dried. The raw product was recrystallized from ethanol.

1-Chloroacetanilides *If*, *Ig*, *Ii*, *Ij*

The mixture, prepared from 0.01 mol substituted aniline and 0.01 mol chloroacetyl chloride in 50 ml of toluene was refluxed for 3 h. After cooling a precipitate of chloroacetanilide was formed, which was removed by filtration.

Substituted 1-(1,2,4-Triazol-1-yl)acetanilides (*IIa* – *IIn*)

The mixture of 0.02 mol corresponding 1-chloroacetanilide, 0.02 mol crystallized 1,2,4-triazole and 0.02 mol (2.8 g) sodium carbonate in 70 ml of acetonitrile was refluxed for 5 h. The solvent was then evaporated almost to dryness and solid residue washed repeatedly with water. Crystallization from ethanol or butanol, respectively, gave pure products.

TABLE II
Spectral data of compounds *IIa* – *IIn*

Compound	IR spectrum (ν, cm^{-1})		^{1}H NMR spectrum (δ, ppm)			
	ν(CO)	ν(NH)	NH[a]	CH_2[a]	H-triazole[a]	
IIa	1 644	3 272	9.35	5.20	8.46	7.92
IIb	1 652	3 264	9.41	5.23	8.46	7.90
IIc	1 656	3 264	9.20	5.23	8.46	7.92
IId	1 680	3 248	9.10	5.36	8.52	8.00
IIe	1 672	3 256	9.16	5.24	8.46	7.93
IIf	1 688	3 208	9.28	5.33	8.49	7.94
IIg	1 672	3 192	9.28	5.12	8.43	7.26
IIh	1 696	3 276	9.45	5.00	8.25	7.47
IIi	1 672	3 248	9.02	5.34	8.53	8.02
IIj	1 680	3 192	9.32	5.31	8.49	7.94
IIk	1 696	3 272	9.82	5.17	8.56	8.00
IIl	1 672	3 224	10.58	5.21	8.58	8.01
IIm	1 692	3 216	9.58	5.19	8.45	7.91
IIn	1 644	3 216	9.70	5.21	8.45	8.02

[a] Singlets.

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