

## Benzothiazole compounds

# XL. Synthesis of 3-(2-alkoxycarbonylethyl)-2-benzothiazolinones and their regulating activity on the growth of *Triticum aestivum* L.

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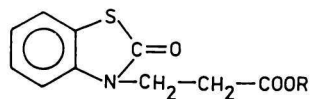
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3-(2-Alkoxycarbonylethyl)-2-benzothiazolinones were prepared by alcoholysis and hydrolysis of 3-(2-cyanoethyl)-2-benzothiazolinones which, in turn, were obtained by addition of 2-hydroxybenzothiazole to acrylonitrile. 3-(2-Ethoxy-, 3-(2-propoxy-, 3-(2-allyloxy-, and 3-(2-butoxycarbonylethyl)-2-benzothiazolinones showed growth-regulating effects towards *Triticum aestivum* L.

Some 3-alkoxycarbonylmethyl-2-benzothiazolinones were found to stimulate plant growth [1—3]. 3-Benzoyloxycarbonylmethyl-2-benzothiazolinone, synthesized by us, showed the highest activity mainly in increasing yields and quality of vegetables, fruits, and cereal crops and in promotion of rooting of woody plants [4—8].

As a continuation of the research in this field, the present paper describes the synthesis of 3-(2-alkoxycarbonylethyl)-2-benzothiazolinones *I—XIII* that are subjects of theoretical considerations and studies of the relationships between the structure and biological activity. The first step in the synthesis was the addition of 2-hydroxybenzothiazole to acrylonitrile in the presence of triethylamine. The product, 3-(2-cyanoethyl)-2-benzothiazolinone, reacted then with alcohols in the presence of dry hydrogen chloride leading to the chlorides of the corresponding iminium esters which afforded free esters by hydrolysis. The alcohols were used in large excess, serving as reaction medium. After pouring the reaction mixture into water, the esters were separated as viscous oils (*II—IV*, *VI—XI*) or solids (*I*, *V*, *XII*, *XIII*). The oily products were extracted with ether. The alcohols insoluble or little soluble in water were extracted by ether together with the esters and were separated from them by distillation *in vacuo*. Since the liquid 3-(2-alkoxycarbonylethyl)-2-benzothiazolinones decom-

Table I  
 Characterization of the prepared 3-(2-alkoxycarbonylethyl)-2-benzothiazolinones



Compound	R	Formula	$M_r$	$w_i(\text{calc.})/\%$				Yield	M.p./ $^{\circ}\text{C}$ Solvent
				$w_i(\text{found})/\%$					
				C	H	N	S	%	
I	CH <sub>3</sub>	C <sub>11</sub> H <sub>11</sub> NO <sub>3</sub> S	237.27	55.74	4.67	5.92	13.52	67	63—65 Methanol
				55.52	4.70	5.81	13.58		
II	C <sub>2</sub> H <sub>5</sub>	C <sub>12</sub> H <sub>13</sub> NO <sub>3</sub> S	251.30	57.42	5.22	5.58	12.77	64	Viscous liquid
				57.24	5.31	5.75	12.48		
III	C <sub>3</sub> H <sub>7</sub>	C <sub>13</sub> H <sub>15</sub> NO <sub>3</sub> S	265.33	58.92	5.70	5.28	12.09	61	Viscous liquid
				58.98	5.77	5.31	12.06		
IV	CH <sub>2</sub> CH=CH <sub>2</sub>	C <sub>13</sub> H <sub>13</sub> NO <sub>3</sub> S	263.31	59.37	4.98	5.32	12.19	62	Viscous liquid
				59.02	4.99	5.09	11.91		
V	CH <sub>2</sub> C≡CH	C <sub>13</sub> H <sub>11</sub> NO <sub>3</sub> S	261.30	59.82	4.24	5.36	12.28	57	51—53 Ether—petroleum ether
				60.05	4.21	5.41	11.98		
VI	C <sub>4</sub> H <sub>9</sub>	C <sub>14</sub> H <sub>17</sub> NO <sub>3</sub> S	279.36	60.27	6.14	5.02	11.49	59	$(\varphi_r = 1:2)$ Viscous liquid
				60.53	6.28	5.00	*11.09		

Table 1 (Continued)

Compound	R	Formula	$M_r$	$w_i(\text{calc.})/\%$ $w_i(\text{found})/\%$				Yield %	M.p./°C Solvent
				C	H	N	S		
VII	i-C <sub>4</sub> H <sub>9</sub>	C <sub>14</sub> H <sub>17</sub> NO <sub>3</sub> S	279.36	60.27	6.14	5.02	11.49	53	Viscous liquid
				60.08	6.30	5.15	11.80		
VIII	i-C <sub>5</sub> H <sub>11</sub>	C <sub>15</sub> H <sub>19</sub> NO <sub>3</sub> S	293.38	61.40	6.52	4.77	10.92	60	Viscous liquid
				61.49	6.43	4.58	10.72		
IX	C <sub>6</sub> H <sub>13</sub>	C <sub>16</sub> H <sub>21</sub> NO <sub>3</sub> S	307.41	62.54	6.88	4.55	10.43	51	Viscous liquid
				62.71	6.65	4.59	10.21		
X	C <sub>8</sub> H <sub>17</sub>	C <sub>18</sub> H <sub>25</sub> NO <sub>3</sub> S	335.46	64.45	7.51	4.17	9.55	71	Viscous liquid
				64.72	7.84	4.02	9.18		
XI	sec-C <sub>8</sub> H <sub>17</sub>	C <sub>18</sub> H <sub>25</sub> NO <sub>3</sub> S	335.46	64.45	7.51	4.17	9.55	68	Viscous liquid
				64.64	7.46	4.09	9.70		
XII	Cyclopentyl	C <sub>15</sub> H <sub>17</sub> NO <sub>3</sub> S	291.37	61.84	5.87	4.80	11.00	55	68 Ether—petroleum ether
				61.94	6.04	4.72	11.33		
XIII	Cyclohexyl	C <sub>16</sub> H <sub>19</sub> NO <sub>3</sub> S	305.39	62.92	6.27	4.58	10.49	55	68—71 Ether
				63.11	6.40	4.39	10.42		

posed to some extent when distilled *in vacuo*, they were purified by column chromatography on silica-gel using benzene as an eluent (Table 1).

This procedure failed in the case of 3-(2-benzyloxycarbonyl)ethyl)-2-benzothiazolinone (*XIV*). This compound was synthesized either from 3-(2-chlorocarbonyl)ethyl)-2-benzothiazolinone and benzyl alcohol in the presence of triethylamine (method *A*) or from 3-(2-carboxyethyl)-2-benzothiazolinone (*XV*) and benzyl alcohol in the presence of sulfuric acid (method *B*). The compound *XV* was obtained by heating of 3-(2-carbamoyl)ethyl)-2-benzothiazolinone in concentrated hydrochloric acid. An attempted preparation of *I—XIII* from esters of  $\beta$ -halo acids and 2-hydroxybenzothiazole was unsatisfactory (yields 10—12%). This reaction, in the presence of a base, proceeded mainly with elimination of hydrogen halide and forming of acrylic acid esters.

Changes in the structure of ester group in the synthesized derivatives have no effect on the position of bands in UV spectra. The spectra, measured in methanol, display strong absorbance at  $\lambda = 208\text{—}214\text{ nm}$  ( $\log(\epsilon/(\text{m}^2\text{ mol}^{-1})) \approx 2.3\text{—}2.5$ ). Further maxima of lower intensity are at  $\lambda = 244\text{ nm}$  ( $\log(\epsilon/(\text{m}^2\text{ mol}^{-1})) \approx 1.7\text{—}1.8$ ), 282 nm and 290 nm ( $\log(\epsilon/(\text{m}^2\text{ mol}^{-1})) \approx 1.4\text{—}1.5$ ). In nonpolar solvents (*e.g.* n-hexane) the UV spectra are bathochromically shifted by 2—4 nm in shortwave region and by 8 nm in longwave region in comparison with the spectra taken in methanol. This shift of absorption maxima can be accounted for by the interaction of lone electron pairs of heteroatoms in the molecules with the polar solvent (methanol). When the lone electron pair of the nitrogen atom in thiazoline ring is in an interaction with methanol, the ability of this pair to conjugate with the  $\pi$ -system decreases and, consequently, a hypsochromic shift of  $\pi \rightarrow \pi^*$  transition occurs. The shift of  $\pi \rightarrow \pi^*$  transitions in the region of  $\lambda = 282\text{—}296\text{ nm}$  can be explained similarly. The comparison of the UV spectrum of 3-(2-methoxycarbonyl)ethyl)-2-benzothiazolinone (*I*) with that of 3-methyl-2-benzothiazolinone (Fig. 1) leads to a conclusion that the effects of both substituents in position 3 on the energy of electron transitions are the same. That means, the effect of the ester group is not transferred through the  $\text{—CH}_2\text{CH}_2\text{—}$  group.

In growth-regulation tests of the synthesized compounds carried out in the concentrations of  $10^{-13}\text{—}10^{-3}\text{ mol dm}^{-3}$  using *Triticum aestivum* L., a good stimulation activity was found for seven derivatives. For three of them, the activity was highly significant (Table 2). The parent compound *XV* is not active but 3-(2-methoxycarbonyl)ethyl)-2-benzothiazolinone (*I*) at  $c = 10^{-7}\text{ mol dm}^{-3}$  showed an increased stimulatory effect by 8.58% with respect to control. Changing the methyl group in the ester moiety for ethyl and propyl ones brings about higher activity. Thus, the ethoxy derivative *II* caused growth stimulation of 13.92% and the corresponding propoxy derivative *III* of 14.13%. Butoxy derivative *VI* is slightly less effective with 10.65% stimulation and the com-

pounds with longer alkyl chains lacked stimulation activity. However, good activity was found for *sec*-octyloxy (*XI*) and cyclopentyloxy (*XII*) derivatives. The highest stimulatory effect, 14.76 %, was attained with allyloxy compound *IV*. On the other hand, propargyloxy derivative *V* was inactive. Curiously enough, for benzyloxy derivative *XIV* no growth-regulating activity was found, in contrast with 3-benzyloxycarbonylmethyl-2-benzothiazolinone [8] that has been proved to be an auxine-like stimulator with effects analogical to those caused by  $\beta$ -indolylacetic,  $\beta$ -indolylbutyric, resp. 2-methyl-4-chlorophenoxyacetic acid [9].

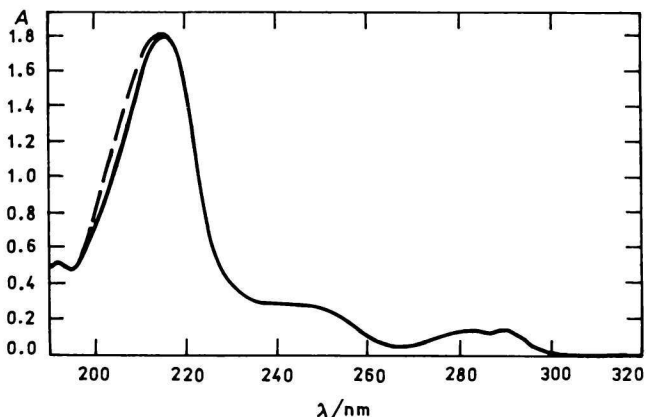


Fig. 1. UV spectrum of 3-(2-methoxycarbonylethyl)-2-benzothiazolinone (---) and 3-methyl-2-benzothiazolinone (—) (methanol,  $l = 1$  mm,  $c = 5 \times 10^{-4}$  mol dm $^{-3}$ ).

## Experimental

Melting points were determined on a Kofler hot-stage apparatus and the analytical data of the synthesized compounds are given in Table 1. UV spectra of compounds ( $c = 10^{-4}$  mol dm $^{-3}$ ) in methanol, resp. n-hexane were measured on an instrument 8452 A (Hewlett—Packard). The growth regulation tests were based on the measurement of elongative growth of primary roots of wheat (*Triticum aestivum* L.) according to a known technique [9].

### 3-(2-Cyanoethyl)-2-benzothiazolinone

The mixture of 2-hydroxybenzothiazole (15.1 g; 0.1 mol), acrylonitrile (7.9 g; 0.15 mol), triethylamine (2.5 g; 0.025 mol), and 120 cm $^3$  of anhydrous methanol was refluxed for 4 h. After cooling, the product was isolated and crystallized from methanol.

Table 2

Growth-regulating activity of the synthesized compounds on *Triticum aestivum* L.

Compound	$\frac{c}{\text{mol dm}^{-3}}$	$\frac{\pm \Delta l}{\text{mm}}$	Stimulation (+) or inhibition (-)		Compound	$\frac{c}{\text{mol dm}^{-3}}$	$\frac{\pm \Delta l}{\text{mm}}$	Stimulation (+) or inhibition (-)	
			%					%	
<i>I</i>	$10^{-13}$	-0.13	-2.79		<i>II</i>	$10^{-13}$	+0.17	+3.53	
	$10^{-}$	-0.01	-0.22			$10^{-11}$	+0.27	+5.61	
	$10^{-9}$	+0.13	+2.78			$10^{-9}$	+0.34	+7.06	
	$10^{-7}$	+0.40	+8.58 <sup>a</sup>			$10^{-7}$	+0.33	+6.86	
	$10^{-}$	+0.27	+5.79			$10^{-}$	+0.67	+13.92 <sup>b</sup>	
	$10^{-}$	-1.44	-30.91			$10^{-}$	-1.47	-30.57	
<i>III</i>	$10^{-13}$	+0.68	+14.13 <sup>b</sup>		<i>IV</i>	$10^{-13}$	+0.03	+0.62	
	$10^{-11}$	+0.21	+4.36			$10^{-11}$	+0.27	+5.61	
	$10^{-9}$	+0.22	+4.57			$10^{-9}$	+0.37	+7.68	
	$10^{-}$	+0.60	+12.47 <sup>b</sup>			$10^{-}$	+0.71	+14.76 <sup>b</sup>	
	$10^{-}$	+0.36	+7.48			$10^{-}$	+0.67	+13.92 <sup>b</sup>	
	$10^{-}$	-1.51	-31.40			$10^{-}$	-1.61	-33.48	
<i>V</i>	$10^{-13}$	-0.19	-4.08		<i>VI</i>	$10^{-13}$	-0.06	-1.53	
	$10^{-11}$	+0.16	+3.43			$10^{-11}$	-0.07	-1.78	
	$10^{-9}$	+0.03	+0.64			$10^{-9}$	-0.19	-4.82	
	$10^{-}$	+0.07	+1.50			$10^{-7}$	+0.10	+2.53	
	$10^{-}$	+0.13	+2.78			$10^{-5}$	+0.42	+10.65 <sup>a</sup>	
	$10^{-}$	-1.61	-34.54			$10^{-3}$	-0.91	-23.10	
<i>VII</i>	$10^{-13}$	-0.34	-6.08		<i>VIII</i>	$10^{-13}$	-0.17	-3.04	
	$10^{-11}$	-0.15	-2.68			$10^{-}$	-0.27	-4.83	
	$10^{-9}$	-0.23	-4.10			$10^{-9}$	-0.15	-2.68	
	$10^{-7}$	-0.10	-1.78			$10^{-7}$	-0.27	-4.83	
	$10^{-5}$	+0.37	+6.60			$10^{-5}$	-0.15	-2.68	
	$10^{-3}$	-1.54	-27.50			$10^{-3}$	-2.39	-42.67	
<i>IX</i>	$10^{-13}$	-0.39	-6.97		<i>X</i>	$10^{-}$	-0.12	-3.05	
	$10^{-11}$	+0.06	+1.60			$10^{-11}$	-0.01	-0.26	

Table 2 (Continued)

Compound	$\frac{c}{\text{mol dm}^{-3}}$	$\frac{\pm \Delta I}{\text{mm}}$	Stimulation (+)	Compound	$\frac{c}{\text{mol dm}^{-3}}$	$\frac{\pm \Delta I}{\text{mm}}$	Stimulation (+)
			or inhibition (-)				or inhibition (-)
			%				%
<i>IX</i>	$10^{-9}$	-0.10	-1.79	<i>X</i>	$10^{-9}$	-0.07	-1.78
	$10^{-7}$	-0.19	-3.40		$10^{-7}$	-0.03	-0.76
	$10^{-5}$	-0.02	-0.36		$10^{-5}$	+0.02	+0.50
	$10^{-3}$	-2.08	-37.14		$10^{-3}$	+0.09	+2.28
<i>XI</i>	$10^{-13}$	+0.34	+8.63 <sup>a</sup>	<i>XII</i>	$10^{-13}$	+0.03	+0.57
	$10^{-11}$	+0.02	+0.50		$10^{-11}$	+0.45	+8.68 <sup>a</sup>
	$10^{-9}$	+0.17	+4.31		$10^{-9}$	-0.15	-2.90
	$10^{-7}$	+0.14	+3.55		$10^{-7}$	+0.33	+6.37
	$10^{-5}$	-0.18	-4.57		$10^{-5}$	+0.19	+3.66
	$10^{-3}$	-0.66	-16.75		$10^{-3}$	-1.50	-28.95
<i>XIII</i>	$10^{-13}$	+0.24	+5.15	<i>XIV</i>	$10^{-13}$	-0.01	-0.21
	$10^{-11}$	-0.07	-1.51		$10^{-11}$	+0.14	+2.86
	$10^{-9}$	+0.20	+4.29		$10^{-9}$	-0.06	-1.23
	$10^{-7}$	+0.11	+2.36		$10^{-7}$	+0.04	+0.81
	$10^{-5}$	+0.19	+4.07		$10^{-5}$	-0.04	-0.82
	$10^{-3}$	+0.33	+7.08		$10^{-3}$	-1.63	-33.34
<i>XV</i>	$10^{-13}$	-0.12	-2.46	IAA	$10^{-13}$	+6.54	+10.28
	$10^{-11}$	-0.36	-7.37		$10^{-11}$	+6.26	+5.56
	$10^{-9}$	-0.24	-0.91		$10^{-9}$	+6.88	+16.02
	$10^{-7}$	-0.10	-2.05		$10^{-7}$	+10.64	+79.42
	$10^{-5}$	-0.12	-2.46		$10^{-5}$	+11.88	+100.33
	$10^{-3}$	-1.84	-37.63		$10^{-3}$	+3.16	+46.72
2,4-D	$10^{-11}$	+4.97	-0.80	CCC	$10^{-13}$	-0.15	-2.78
	$10^{-9}$	+5.04	+0.59		$10^{-11}$	-0.07	-1.30
	$10^{-7}$	+5.06	+0.99		$10^{-9}$	-0.12	-2.22
	$10^{-5}$	+7.57	+51.09		$10^{-7}$	-0.30	-5.55
	$10^{-3}$	+3.00	-40.12		$10^{-5}$	-0.07	-1.30
				$10^{-3}$	-1.75	-32.32	

<sup>a</sup>) Significant; <sup>b</sup>) highly significant. IAA —  $\beta$ -indolylacetic acid, 2,4-D — 2,4-dichlorophenoxyacetic acid, CCC — (2-chloroethyl)tri-methylammonium chloride.

Yield = 18 g (88 %), m.p. = 117—119 °C. For  $C_{10}H_8N_2OS$  ( $M_r = 204.25$ )  $w_i$ (calc.): 58.87 % C, 3.95 % H, 13.73 % N, 15.71 % S;  $w_i$ (found): 58.71 % C, 4.08 % H, 13.88 % N, 15.59 % S. UV spectrum,  $\lambda_{\max}/nm$  ( $\log(\epsilon/(m^2 mol^{-1}))$ ): 288 (2.39), 282 (2.40), 244 (2.70), 220 (3.23).

### *3-(2-Alkoxy-carbonyl-ethyl)-2-benzothiazolinones I—XIII*

The solution of 3-(2-cyanoethyl)-2-benzothiazolinone (10.2 g; 0.05 mol) in 50 cm<sup>3</sup> of an appropriate alcohol was heated on a boiling water bath and anhydrous hydrogen chloride was introduced through a capillary for 5 h. The mixture was allowed to stand at room temperature for 12 h, then poured into cold water and oily precipitate was extracted with ether. The alcohols insoluble in water were extracted together with the synthesized compounds. The extract was dried over sodium sulfate, ether and alcohol were evaporated and the liquid residue (*II—IV*, *VI—XI*) was purified on a column of silica gel L 100/250 (Lachema, Brno) using benzene as an eluent. Solid residue (*I*, *V*, *XII*, *XIII*) was purified by crystallization (Table 1).

### *3-(2-Carbamoyl-ethyl)-2-benzothiazolinone*

3-(2-Cyanoethyl)-2-benzothiazolinone (4 g; 0.02 mol) was added in small portions to concentrated sulfuric acid (30 cm<sup>3</sup>) with stirring. After dissolving, the mixture was left to stand at room temperature for 24 h, then poured on crushed ice. The crystalline product was filtered off, washed with small amount of ice-cold water and crystallized from ethanol. Yield = 4 g (90 %), m.p. = 188—189 °C. For  $C_{10}H_{10}N_2O_2S$  ( $M_r = 222.26$ )  $w_i$ (calc.): 54.04 % C, 4.53 % H, 12.50 % N, 14.42 % S;  $w_i$ (found): 54.12 % C, 4.40 % H, 12.54 % N, 14.34 % S. UV spectrum,  $\lambda_{\max}/nm$  ( $\log(\epsilon/(m^2 mol^{-1}))$ ): 290 (2.41), 282 (2.41), 244 (2.73), 220 (3.29).

### *3-(2-Carboxyethyl)-2-benzothiazolinone (XV)*

3-(2-Carbamoyl-ethyl)-2-benzothiazolinone (4.4 g; 0.02 mol) was added to concentrated hydrochloric acid (15 cm<sup>3</sup>) and the mixture was heated at reflux temperature for 30 min. After cooling, the crystalline product was filtered off, washed with cool water and recrystallized from acetone. Yield = 4.2 g (94 %), m.p. = 140—141 °C. For  $C_{10}H_9NO_3S$  ( $M_r = 223.25$ )  $w_i$ (calc.): 53.86 % C, 4.07 % H, 6.28 % N, 14.37 % S;  $w_i$ (found): 53.98 % C, 3.91 % H, 6.39 % N, 14.25 % S. UV spectrum,  $\lambda_{\max}/nm$  ( $\log(\epsilon/(m^2 mol^{-1}))$ ): 290 (2.43), 282 (2.44), 244 (2.75), 218 (3.35).

### *3-(2-Chlorocarbonyl-ethyl)-2-benzothiazolinone*

The compound *XV* (4.9 g; 0.022 mol) and thionyl chloride (8.4 g; 0.07 mol) were dissolved in 50 cm<sup>3</sup> of anhydrous benzene. The mixture was stirred vigorously and



refluxed for 3 h. Unreacted thionyl chloride and one third of benzene were distilled off and the residue was cooled. The crystalline product was filtered off, washed with anhydrous cyclohexane and dried in a desiccator *in vacuo*. The product decomposes when exposed to humid air. Yield = 4.7 g (88 %), m.p. = 63—65 °C. For  $C_{10}H_8ClNO_2S$  ( $M_r = 241.69$ )  $w_i(\text{calc.})$ : 49.69 % C, 3.33 % H, 5.79 % N, 13.26 % S, 14.66 % Cl;  $w_i(\text{found})$ : 49.30 % C, 3.76 % H, 5.62 % N, 13.31 % S, 14.75 % Cl.

### 3-(2-Benzoyloxycarbonylethyl)-2-benzothiazolinone (XIV)

*Method A.* The solution of benzyl alcohol (6.4 g; 0.06 mol) and triethylamine (5 g; 0.05 mol) in 20 cm<sup>3</sup> of acetone was added dropwise to the stirred solution of 3-(2-chloro-carbonylethyl)-2-benzothiazolinone (12 g; 0.05 mol) in 30 cm<sup>3</sup> of anhydrous acetone. Afterwards, the mixture was refluxed for 5 h, cooled and water (75 cm<sup>3</sup>) was added. Viscous oil was separated and aqueous layer was extracted with ether. The etheric extract was combined with the viscous oil, dried over sodium sulfate and the solvent was evaporated. The oily product was purified on a column of silica gel using benzene as an eluent. Yield = 68 % (viscous liquid). For  $C_{17}H_{15}NO_3S$  ( $M_r = 313.37$ )  $w_i(\text{calc.})$ : 65.23 % C, 4.83 % H, 4.47 % N, 10.24 % S;  $w_i(\text{found})$ : 64.98 % C, 4.97 % H, 4.24 % N, 10.07 % S.

*Method B.* Benzyl alcohol (10.8 g; 0.1 mol) and three drops of concentrated sulfuric acid were added to the solution of XIV (11.15 g; 0.05 mol) in 50 cm<sup>3</sup> of benzene. The mixture was refluxed using Dean—Stark trap until no more water was separated. The reaction mixture was cooled, washed with 10 % solution of potassium carbonate and with water and dried over sodium sulfate. Benzene was evaporated and the product was purified as in method A. Yield = 63 %.

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