

# Synthesis of 2-aryl-4-phthalidyldien-1,3-oxazolin-5-ones

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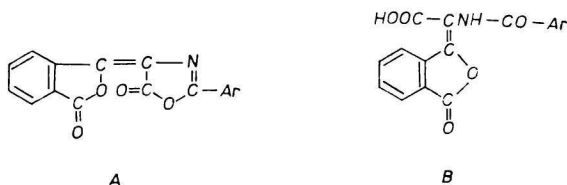
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Some 2-aryl-4-phthalidyldien-1,3-oxazolin-5-ones were prepared by a Perkin condensation of phthalic anhydride with the corresponding aroylglycine in the presence of acetic anhydride and sodium acetate.

Although phthalic anhydride has been used successfully as a carbonyl component in Perkin reaction [1–4], its reaction with hippuric acid and its derivatives has not been thoroughly investigated.

As reported by *Erlenmeyer* [5], phthalic anhydride condenses with hippuric acid to give a product for which he presumed one of the structures *A* or *B* in Scheme 1 ( $\text{Ar} = \text{C}_6\text{H}_5$ ), but he did not establish either of them.



In this investigation we report the synthesis of some new 2-aryl-4-phthalidyldien-1,3-oxazolin-5-ones (*I–IX*; Table 1) in good yields. The reaction was carried out by warming phthalic anhydride and the corresponding aroylglycine in the presence of acetic anhydride containing freshly fused sodium acetate.

The products obtained (*I–IX*) were characterized both by analysis and by i.r. measurements, which showed stretching frequencies at about 1790 and 1640  $\text{cm}^{-1}$  characteristic of the carbonyl group of  $\gamma$ -lactone and  $\text{—C=N—}$  respectively. In addition, these products were found to be insoluble in dilute sodium hydrogen carbonate solution which indicates the absence of a free carboxylic group. The products do not give any colouration with iron(III) chloride solution which showed the absence of  $\text{—NH—CO—}$  group. These facts prove unequivocally that the reaction products were 2-aryl-4-phthalidyldien-1,3-oxazolin-5-ones. No other products of *B* structure were obtained.

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Table 1

Characterization of the prepared 2-aryl-4-phthalidyliden-1,3-oxazolin-5-ones

No.	Aryl	Formula	M	Calculated/found				Yield [%]	M.p. [°C]	Colour
				% C	% H	% N	% X			
I	Phenyl	C <sub>17</sub> H <sub>9</sub> NO <sub>4</sub>	291.23	70.11	3.12	4.81		82	247	Yellow
				70.08	3.10	4.75				
II	<i>o</i> -Chloro-phenyl	C <sub>17</sub> H <sub>8</sub> ClNO <sub>4</sub>	325.67	62.69	2.48	4.30	10.86	90	265	Yellow
				62.70	2.41	4.27	10.75			
III	<i>p</i> -Chloro-phenyl	C <sub>17</sub> H <sub>8</sub> ClNO <sub>4</sub>	325.67	62.69	2.48	4.30	10.86	79	287	Yellow
				62.66	2.48	4.33	10.82			
IV	<i>o</i> -Iodo-phenyl	C <sub>17</sub> H <sub>8</sub> INO <sub>4</sub>	417.12	48.95	1.94	3.36	30.42	85	260	Yellow
				48.89	1.86	3.32	30.36			
V	<i>o</i> -Tolyl	C <sub>18</sub> H <sub>11</sub> NO <sub>4</sub>	305.26	70.82	3.64	4.59		79	215	Golden yellow
				70.70	3.61	4.53				
VI	<i>m</i> -Tolyl	C <sub>18</sub> H <sub>11</sub> NO <sub>4</sub>	305.26	70.82	3.64	4.59		82	242	Brown
				70.75	3.56	4.55				
VII	<i>p</i> -Tolyl	C <sub>18</sub> H <sub>11</sub> NO <sub>4</sub>	305.26	70.82	3.64	4.59		84	228	Orange
				70.82	3.58	4.57				
VIII	Benzyl	C <sub>18</sub> H <sub>11</sub> NO <sub>4</sub>	305.26	70.82	3.64	4.59		70	215	Yellow
				70.90	3.58	4.50				
IX	<i>p</i> -Methoxy-phenyl	C <sub>18</sub> H <sub>11</sub> NO <sub>5</sub>	321.25	67.29	3.46	4.36		85	220	Orange
				67.30	3.40	4.32				

### Experimental

Aroylglycines were prepared according to the Schotten—Baumann method of benzoylation [6]. Analytical data were determined in a microanalytical unit, Cairo University. Infrared spectra were recorded on an SP 1200 Pye-Unicam spectrophotometer. Melting points were not corrected.

#### *2-Aryl-4-phthalidyliden-1,3-oxazolin-5-ones (I—IX)*

A mixture of phthalic anhydride (0.01 mole) and aroylglycine (benzoyl-, *o*-chlorobenzoyl-, *p*-chlorobenzoyl-, *o*-iodobenzoyl-, 2-methylbenzoyl-, 3-methylbenzoyl-, 4-methylbenzoyl-, phenacetyl-, and 4-methoxybenzoylglycine, respectively; 0.01 mole) in acetic anhydride (20 ml) containing freshly fused sodium acetate (0.5 g) was heated on a water bath at 100°C for 2 hrs. The reaction mixture was cooled and the precipitated solid was collected by filtration, washed with 10% sodium hydrogen carbonate solution (50 ml), water (50 ml), and ethanol (20 ml). Recrystallization of the products was from dioxan, compound *I* from acetic acid. Characterization of the products is in Table I.

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