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PREPARATION OF ISATOIC ANHYDRIDE FROM PHTHALIMIDE

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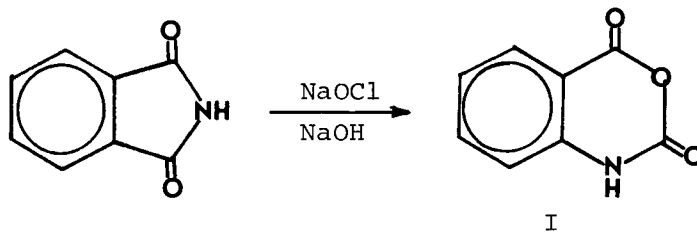
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PREPARATION OF ISATOIC ANHYDRIDE FROM PHTHALIMIDE

Submitted by Y. R. Rao,* M. Bapuji and S. N. Mahapatra
(5/13/81)

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The preparation of isatoic anhydride(I) from anthranilic acid¹ in 75% yields has the disadvantage of using poisonous phosgene and the product has to be collected in two or three lots. Its manufacture from phthalimide is described without the exact details.² We now describe in detail a modified procedure for its preparation.



EXPERIMENTAL

To a stirred mixture of phthalimide (147 g, 1 mole), crushed ice (400 g) and water (250 ml) in a 5 l beaker was slowly added through a dropping funnel a solution of 40 g of sodium hydroxide in 350 ml of water; most of the phthalimide dissolved. To this solution of sodiophthalimide at 0°, was gradually added 950 ml of 1M sodium hypochlorite³ over a period of 20 min. with stirring; the reaction mixture was maintained at 4-6°. After an additional 15 min., the solution was acidified with conc. hydrochloric acid to adjust the pH to 6 and the slurry stirred for 1.5 hr. The precipitated solid was collected, washed with water until the washings were colorless and dried at 45-50° to give 136-140 g (85-88%) isatoic anhy-

dride, mp. 240-242^o (dec.) as a creamy white solid. (Assay by titrimetry: min 98%).

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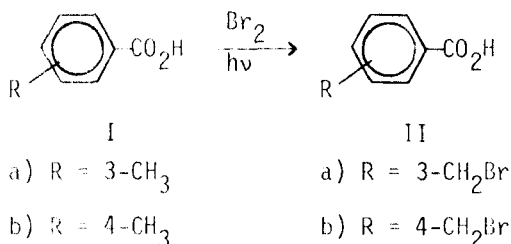
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HIGH-YIELD, ONE STEP PHOTOCHEMICAL SYNTHESIS
OF 3- AND 4-BROMOMETHYLBENZOIC ACIDS

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The few reported^{1,2} procedures for the preparation of 3- and 4-methylbenzoic acids (II) utilize relatively expensive or unavailable reagents or starting materials or are patents^{3,4} for which few details are given. We now report a simple one-step, good yield procedure for the preparation of IIa and IIb.



EXPERIMENTAL

3-Bromomethylbenzoic Acid (IIa). 4-Methylbenzoic acid (12.2 g, 0.090