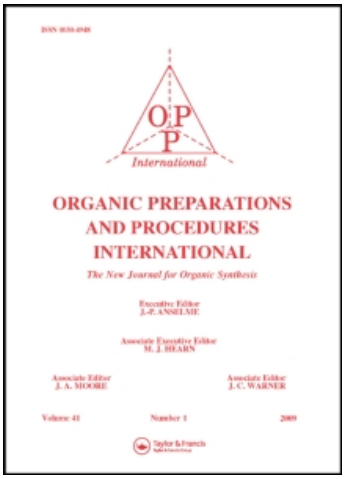

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PREPARATION OF ISATOIC ANHYDRIDE FROM PHTHALIMIDE

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PREPARATION OF ISATOIC ANHYDRIDE FROM PHTHALIMIDE

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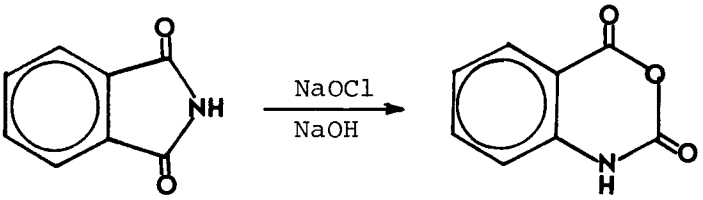
The preparation of isatoic anhydride(I) from anthranilic acid[1] in 75% yields has the disadvantage of using poisonous phosgene and the product has to be collected in two or three lots. Its manufacture from phthalimide is described without the exact details.[2] We now describe in detail a modified procedure for its preparation.



I

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 hypochlorite[3] over a period of 20 min. with stirring; the reaction mixture was maintained at $4\text{-}6^{\circ}$. 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 $45\text{-}50^{\circ}$ to give 136-140 g (85-88%) isatoic anhy-

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

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

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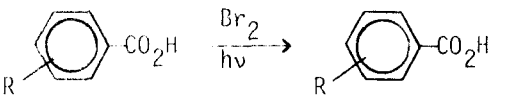
The few reported[1,2] procedures for the preparation of 3- and 4-methylbenzoic acids (II) utilize relatively expensive or unavailable reagents or starting materials or are patents[3,4] for which few details are given. We now report a simple one-step, good yield procedure for the preparation of IIa and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



I

a) R = 3-CH_3

b) R = 4-CH_3

II

a) R = 3-CH_2Br

b) R = 4-CH_2Br

EXPERIMENTAL

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