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Yuichi Kanaoka : Application of the Robinson Dehydrogenation Reaction. V[1] (Supplement to Part I[2]). Synthesis of 2,3-Dimethoxy-5-methyl-10b,11-dihydro-6*H*-indolo[2,1-*a*]isoindolium Iodide.

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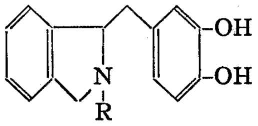
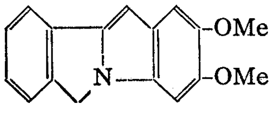
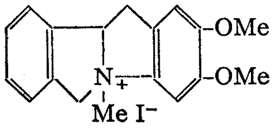
In Part I[2] of this series, the synthesis of indoloisoindole derivative (III) from isoindoline derivative (I) was reported, whereby the N-methyl compound (II) failed to give any definite product.



(I) R=H
(II) R=Me



(III)



(IV)

Based on improved treatment, (II) was cyclized oxidatively to give the corresponding indolium salt (IV) after methylation, in a good yield.

Thus, in the case of the isoindoline, like other amines described in the earlier papers,[3] it was found that the tertiary amine (II) gave the indolium salt, while the secondary amine (I) gave the indole derivative as the result of further oxidation of the initially formed base.

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Experimental

Oxidation of (II); 2,3-Dimethoxy-5-methyl-10b,11-dihydro-6*H*-indolo[2,1-*a*]isoindolium Iodide (IV)—A mixture of (II) (0.2 g.), EtOH (40 cc.), and KOAc (0.05 g.) was warmed until a clear solution resulted. A small amount of undissolved material was filtered off, the filtrate was oxidized with chloranil (0.07 g.) and suspended in EtOH (100 cc.) as usual. A reddish brown coloration appeared on addition of reagent, then disappeared rapidly (positive). Addition was completed in 20 min. and the solution became reddish. After allowing it to stand for 10 min., dil. HCl (5 cc.) was added and the color faded instantly. The residue, obtained by filtration followed by evaporation, was extracted with warm water (50 cc.) and evaporated *in vacuo*. The residual powder was dissolved in 33% KOH (2 cc.), shaken vigorously with Me_2SO_4 (2 cc.), then 33% K_2CO_3 (2 cc.) and Me_2SO_4 (1 cc.) were added, and shaken again.

Finally, the mixture was warmed briefly with 33% K_2CO_3 (5 cc.), taken up in $CHCl_3$ after cooling, and then extracted with water (20 cc., 3 times). To the filtered water layer KI (1 g.) was added, and the mixture was stored in a refrigerator overnight. The separated solid was collected and purified from MeOH-Et_2O, forming almost colorless prisms, m.p. 245~247°(decomp.). Yield, 75 mg. or 63%. *Anal.* Calcd. for $C_{18}H_{20}O_2NI$: C, 52.82; H, 4.92; N, 3.42. Found : C, 52.8; H, 4.85; N, 3.6.

(IV) (2 mg.) was heated at about 250° (bath temp.) for a few mins. and dissolved in EtOH (0.5 cc.), which developed a violet color in Ehrlich's test.

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[1] Part IV : Y. Kanaoka : This Bulletin, 7, 597(1959).

[2] Part I : S. Sugasawa, Y. Kanaoka : *Ibid.*, 3, 266(1955).

[3] Part II : Y. Kanaoka : *Ibid.*, 7, 589(1959).