

REISSERT COMPOUND STUDIES. XXIX.

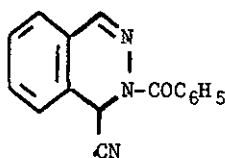
AN IMPROVED SYNTHESIS OF THE PHTHALAZINE REISSERT COMPOUND
BY PHASE TRANSFER CATALYSIS

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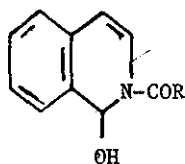
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Treatment of phthalazine with benzoyl chloride and potassium cyanide gives in addition to the Reissert compound, the pseudo-base. Use of a phase transfer catalyst suppresses the formation of the pseudo-base.

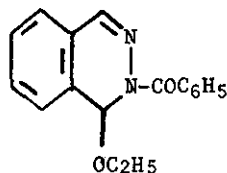
We reported earlier¹ that phthalazine, benzoyl chloride, and potassium cyanide in methylene chloride-water reacted to give the Reissert compound I. In connection with other work in progress in this laboratory we had need to synthesize additional quantities of I. We found the yields and purity of I to be highly inconsistent. The product I was contaminated with the pseudo-base and in some cases the pseudo-base was the major product. Pseudo-bases of the type II have been previously reported²⁻⁵ in the prep-



I



II



III

aration of Reissert compounds from isoquinolines.

When the crude reaction mixture from phthalazine, benzoyl chloride and potassium cyanide was recrystallized from ethanol we obtained, in addition

to I, substantial quantities of III, M.p. 91° (calcd. for C₁₇H₁₆N₂O₂: C, 72.84; H, 5.75; N, 9.99. Found: C, 72.83; H, 5.75; N, 9.98.). The conversion of a pseudo-base to an ethoxy compound on recrystallization from ethanol had been previously noted.^{3,4}

In order to improve the transfer of the cyanide ion to the organic medium we investigated the effect of adding a phase transfer catalyst in the preparation of I. Addition of a catalytic amount of benzyltriethylammonium chloride to the reaction of phthalazine, benzoyl chloride, and potassium cyanide in methylene chloride-water gave good yields of I (over 60%) uncontaminated by III. Similar results have recently been reported.⁶

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