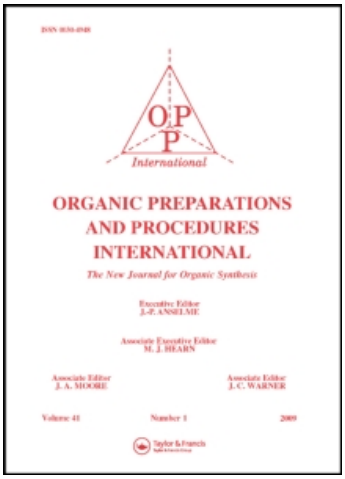

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AN IMPROVED SYNTHESIS OF PHTHALAZINE REISSERT COMPOUND

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AN IMPROVED SYNTHESIS OF PHTHALAZINE REISSERT COMPOUND

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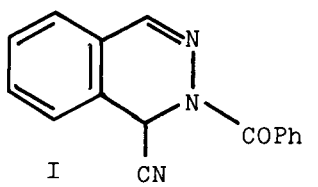
The preparation of Reissert compound I by the reaction of phthalazine with benzoyl chloride and potassium cyanide in methylene chloride-water,[1] was later shown[2] to give highly inconsistent results, though the addition of a phase-transfer catalyst gave a 60% yield of I.[2,3] Compound I can be obtained in 88% yield from the reaction of phthalazine, benzoyl chloride and trimethylsilyl cyanide in the presence of a catalytic amount of aluminium chloride; this method has been applied to the synthesis of various Reissert compounds of quinolines and isoquinolines.[4]



I

EXPERIMENTAL

Trimethylsilyl cyanide (0.16 ml) was injected through a rubber septum to a stirred solution of phthalazine (0.1813 g, 1.39 mmole) and a catalytic amount of anhydrous aluminium chloride in methylene chloride (20 ml). The mixture was allowed to stir for 5 min at RT then freshly distilled benzoyl chloride (0.1630 g, 1.16 mmole) was added and the mixture was stirred overnight. The reaction mixture was then passed through a short column of silica gel and eluted with methylene chloride to give

0.2664 g. (88%) of N-benzoyl-1-cyanophthalazine, mp. 165-165.5°, lit.[1] mp. 163-164°; IR (nujol mull): 1645 cm^{-1}; NMR ($CDCl_3$): δ 6.7 (s, 1H, -CHCN), 7.35-7.90 (m, 9H, ArH), 7.78 (s, 1H, HC=N); MS: m/e 261 (P^+).

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