SYNTHESIS OF 7-METHYL-9,10-DIMETHOXYBENZIMIDAZO-[3,2-b]ISOQUINOLINIUM PERCHLORATE

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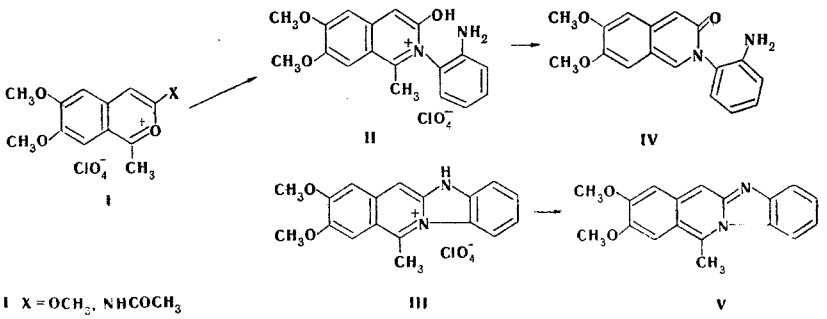
We have found that N-(2-aminophenyl)isoquinolinium (II) and benzimidazo[3,2-b]isoquinolinium (III) perchlorates are formed by the action of o-phenylenediamine on 2-benzopyrylium salts (I).



The direction of the reaction depends on the solvent used in the reaction and the temperature. Thus when I is mixed with o-phenylenediamine in acetone, 1-methyl-2-(2-aminophenyl)-3-hydroxy-6,7-dimethoxyisoquinolinium perchlorate (II, X = OCH_3) is formed in 45% yield and II (X = $NHCOCH_3$) is obtained in 73% yield as yellow prisms with mp 306-307° (reprecipitation from dimethylformamide by the addition of water). IR spectrum (mineral oil): 3550, 3490, 1638, 1560, 1525, 1492, and 1100 cm^{-1}. A hypsochromic shift of the absorption maximum, which we ascribed to the hydroxy (oxo) group, on passing from methanol [λ_{max} 420 nm (log ε 3.63)] to dichloroethane [λ_{max} 405 nm (log ε 3.54)] is observed in the UV spectra of methanol and dichloroethane solutions of II. This constitutes evidence that II exists in two tautomeric forms. The tautomeric equilibrium in solutions of II is probably shifted to favor the hydroxy form due to the formation of an intramolecular hydrogen bond. Base IV was obtained as yellow prisms with mp 124-125° (from water) by the action of excess ammonia on isoquinolinium salt II.

7-Methyl-9,10-dimethoxybenzimidazo[3,2-b]isoquinolinium perchlorate (III) was obtained by heating salt I with o-phenylenediamine in ethanol for 2 h. Workup gave yellow prisms with mp 316-317° (from nitromethane) in 67% yield. IR spectrum (mineral oil): 3215 (m), 1625, 1560, and 1115 cm^{-1}. The N-methyl derivative is formed in the reaction of III with an excess of an ether solution of diazomethane; the yield of colorless prisms with mp 254-255° (from nitromethane) was 83%.

Base V was obtained as yellow prisms with mp 178-179° (from ethanol). The results of elementary analysis for all of the compounds obtained were in agreement with the calculated values.

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