NEW SYNTHESIS OF ACRIDINE DERIVATIVES

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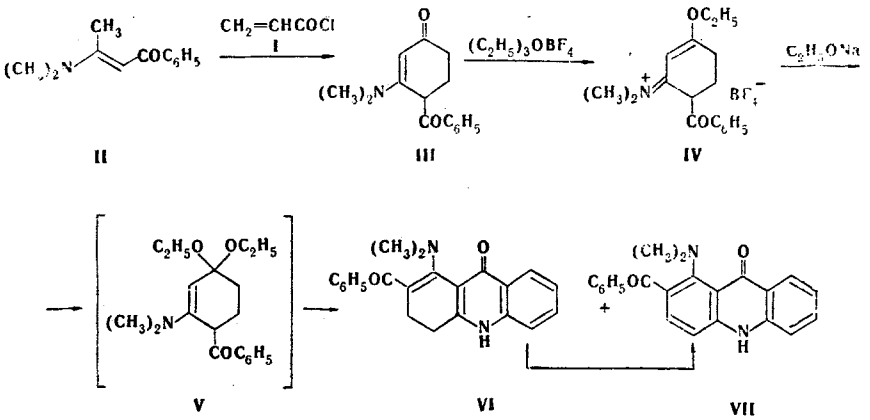
It is known that enamines react with acryloyl chloride (I) to give substituted cyclohexanones [1]. From 1-phenyl-4-dimethylaminobut-3-en-2-one (II) we have similarly obtained 3-dimethylamino-4-benzoyl-Δ^2-cyclohexenone (III), ethylation of which leads to tetrafluoroborate IV. The latter was converted to diethylacetal V, which was subjected to condensation with ethyl anthranilate to give a mixture of acridines (VI and VII), as evidenced by the PMR spectrum, in which signals of protons of an NMe_2 group (~2.90 ppm) and aromatic and NH protons (7.09-8.10 ppm) and a multiplet at 4.95-5.26 ppm with an intensity of two proton units were observed, i.e., the VI:VII ratio is 1:1. Molecular-ion peaks with m/e 342 and 344 are observed in the mass spectrum.



A solution of 7.5 g (0.83 mole) of chloride I in 200 ml of benzene was added dropwise in the course of 5.5 h to a refluxing solution of 14.4 g (0.076 mole) of enamine II in 630 ml of anhydrous benzene, 31.3 g of triethylamine was added, and the mixture was refluxed for 30 min. The solid material was removed by filtration and extracted with chloroform to give 0.7 g of ketone III. The benzene was removed by distillation, and the residue was triturated with ethyl acetate to give 7.4 g of III. The ethyl acetate was filtered through a layer of silica gel, after which elution with chloroform gave another 0.9 g of III. The overall yield of III, with mp 193-194°C (from acetone), was 9 g (48%). A solution of 3.5 g of Et_3OBF_4 in 15 ml of dry CH_2Cl_2 was added to a solution of 4.1 g of ketone III in 40 ml of CH_2Cl_2, and the mixture was stirred at 20°C for 6 h. The solvent was then removed by distillation to give 6 g of tetrafluoroborate IV with mp 118-119°C (from ethyl acetate). A 10-g sample of IV was added at 0°C to a solution of sodium ethoxide (from 0.53 g of Na and 20 ml of alcohol), and the mixture was allowed to stand for 30 min. It was then filtered, and the alcohol was removed by vacuum distillation. Dry toluene (40 ml) and 3.4 g of ethyl anthranilate were added to the residue, and the mixture was refluxed for 4 h. The toluene was removed by distillation to give 1.25 g of a mixture of VI and VII. Nitrobenzene (10 ml) was added to the mixture, and the mixture was refluxed for 2 h. Workup gave 7-benzoyl-8-N,N-dimethylamino-9-acridone (VII) with mp > 300°C (from dimethylformamide). The PMR and mass spectra were in agreement with structure VII. The results of elementary analysis of the compounds obtained were in agreement with the calculated values.

LITERATURE CITED

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