SYNTHESIS OF 4H-CYCLOPENTA[k,l,m]PHENANTHRIDINE-5,9-DIONE

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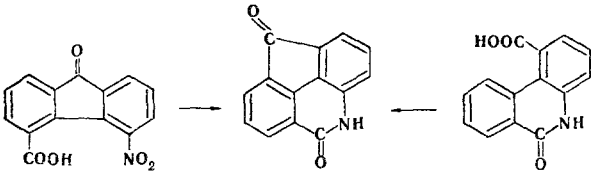
Reduction of the nitro group in 5-nitrofluorenone-4-carboxylic acid (I) leads to simultaneous closing of a lactam ring to give the previously undescribed 4H-cyclopenta[k, l,m]phenanthridine-5,9-dione system (II), which was difficult to anticipate in view of the strain arising here. One can also arrive at this compound by cyclization of phenanthridone-1-carboxylic acid by the action of concentrated sulfuric acid.



I II III

A mixture of 2 g of fluorenonenitro carboxylic acid I and 10 g of sodium hydrosulfite was refluxed in 60 ml of 50% ethanol for 1 h, after which it was acidified with hydrochloric acid and filtered. The solid was crystallized from aqueous dimethylformamide (DMF) to give yellow rhombic crystals with mp > 340° in 70% yield. IR spectrum (UR-20, KBr), cm^{-1}: 2900-3200 (NH), 1724 (CO), 1666 (CO—NH), 1624, 1597, 1500, 1450, 1420, 1382, 1320, 1300, 1260, 1200, 1016, 900, 760, 730, and 693. Found: C 76.0; H 3.4; N 6.4%. $C_{14}H_7NO_2$. Calculated: C 76.0; H 3.2; N 6.3%. Thin-layer chromatography was carried out with Silufol plates with a 25% ammonium hydroxide—dioxane system (1:4); R_f 0.84.

A 1-g sample of phenanthridone-1-carboxylic acid III was heated with 25 ml of concentrated sulfuric acid at 150° for 1 h, after which the mixture was poured over ice, and the precipitate was worked up to give II.

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