METHOD FOR THE PREPARATION OF 5,6,7,8-TETRAHYDRO-8-OXO-1,3-DIOXOLO[4,5-g]QUINOLINE

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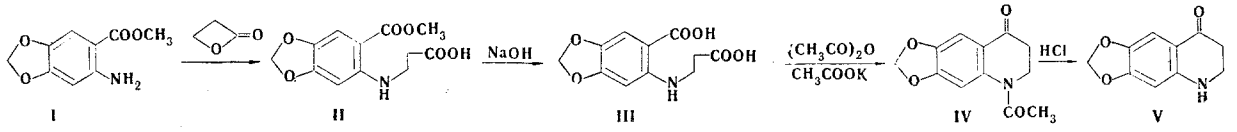
In developing our research on the synthesis of hydrogenated quinolines by cyclization of N-β-carboxyethylanthranilic acids [1], we have accomplished the synthesis of 5,6,7,8-tetrahydro-8-oxo-1,3-dioxolo[4,5-g]-quinoline (IV) from methyl 6-aminopiperonylate (I).

EXPERIMENTAL

Methyl 6-(β-Carboxyethylamino)piperonylate (II). This compound was obtained in 83% yield as colorless crystals with mp 167-168°C (from acetic acid) by the reaction of I with β-propiolactone in acetonitrile. Found: C 53.8; H 4.9; N 5.1%. $C_{12}H_{13}NO_6$. Calculated: C 53.9; H 4.9; N 5.2%.



I II III IV V

6-(β-Carboxyethylamino)piperonylic Acid (III). This compound was obtained in 91% yield as colorless crystals with mp 170-172° (with decomposition, purified by reprecipitation) by alkaline hydrolysis of II. Found: C 52.1; H 4.1; N 5.5%. $C_{11}H_{11}NO_6$. Calculated: C 52.2; H 4.3; N 5.5%.

5-Acetyl-5,6,7,8-tetrahydro-8-oxo-1,3-dioxolo[4,5-g]quinoline (IV). This compound was obtained in 47% yield as light-yellow crystals with mp 153-155° (from benzene and then from alcohol) by cyclization of III in acetic anhydride in the presence of potassium acetate. IR spectrum: ν_{CO} 1665 cm^{-1} (mineral oil suspension, with a UR-20 spectrophotometer). UV spectrum, λ_{max}, nm (log ε): 254 (4.27), 280 (3.78), 338 (3.67) (in alcohol, with an SF-4A spectrophotometer). Found: C 61.5; H 4.7; N 6.2%. $C_{12}H_{11}NO_4$. Calculated: C 61.8; H 4.8; N 6.1%.

5,6,7,8-Tetrahydro-8-oxo-1,3-dioxolo[4,5-g]quinoline (V). This compound was obtained in 71% yield as light-yellow crystals with mp 176-177° (from alcohol) by acid hydrolysis of IV. IR spectrum: ν_{NH} 3305 cm^{-1}, ν_{CO} 1640 cm^{-1}. UV spectrum, λ_{max}, nm (log ε): 246 (4.24), 388 (3.80). Found: C 63.2; H 4.9; N 7.2%. $C_{10}H_9NO_3$. Calculated: C 62.8; H 4.8; N 7.3%. The p-nitrophenylhydrazone of V was obtained as dark-cherry-red crystals with mp 239-241° (dec., from aqueous alcohol). Found: N 17.1%. $C_{16}H_{14}N_4O_4$. Calculated: N 17.2%.

LITERATURE CITED

1. A. F. Bekhli and F. S. Mikhailitsyn, Khim. Geterotsikl. Soedin., 235 (1971).

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