SYNTHESIS OF HYDROGENATED 6,6'-DIQUINOLYL DERIVATIVES

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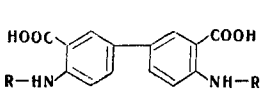
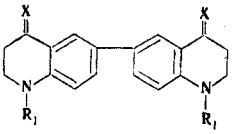
We have found a new route to the synthesis of hydrogenated 6,6'-diquinolyl derivatives. The synthesis consists in cyclization of 4,4'-di(β-dicarboxyethylamino)diphenyl-3,3'-dicarboxylic acid (III). Acid III was obtained from 4,4'-diaminodiphenyl-3,3'-dicarboxylic acid (I) by cyanoethylation and subsequent hydrolysis of the resulting 4,4'-di(β-cyanoethylamino)diphenyl-3,3'-dicarboxylic acid (II).



I–III



IV–VII

I R=H; II R=$(CH_2)_2CN$; III R=$(CH_2)_2COOH$; IV R_1=$COCH_3$, X=O; V R_1=$COCH_3$, X=N–NH_2; VI R_1=H, X=O; VII R_1=H, X=N–NH_2

EXPERIMENTAL

4,4'-Di(β-cyanoethylamino)diphenyl-3,3'-dicarboxylic Acid (II). This compound had mp 256-258° (dec., from acetic acid). Found: C 63.6; H 4.9; N 14.7%. $C_{20}H_{18}N_4O_4$. Calculated: C 63.5; H 4.8; N 14.8%.

4,4'-Di(β-carboxyethylamino)diphenyl-3,3'-dicarboxylic Acid (III). This compound had mp 240-241° (dec., from acetic acid). Found: C 57.5; H 4.9; N 6.7%. $C_{20}H_{20}N_2O_8$. Calculated: C 57.7; H 4.8; N 6.7%.

1,1'-Diacetyl-1,1', 2,2', 3,3', 4,4'-octahydro-4,4'-dioxo-6,6'-diquinolyl (IV). This compound was obtained in 40% yield by cyclization of III in acetic anhydride in the presence of potassium acetate. The light-yellow crystals had mp 248-250° (from aqueous dimethylformamide). Found: C 70.7; H 5.5; N 7.4%. $C_{22}H_{20}N_2O_4$. Calculated: C 70.2; H 5.4; N 7.4%.

Dihydrazone of IV (V). This compound was obtained by heating IV with hydrazine hydrate in ethylene glycol and had mp 330-332° (dec., from ethylene glycol). Found: N 20.4%. $C_{22}H_{24}N_6O_2$. Calculated: N 20.8%.

1,1', 2,2', 3,3', 4,4'-Octahydro-4,4'-dioxo-6,6'-diquinolyl (VI). This compound was isolated after hydrolysis of IV in acidic media and had mp 320-322° (from ethylene glycol). Found: C 76.2; H 5.6; N 9.5%. $C_{18}H_{16}N_2O_2$. Calculated: C 76.6; H 5.7; N 9.6%. The dihydrochloride of VI had mp ~332° (dec.). Found: Cl 19.2; N 7.9%. $C_{18}H_{16}N_2O_2 \cdot 2HCl$. Calculated: Cl 19.4; N 7.7%.

Dihydrazone of VI (VII). This compound was obtained by the method used to prepare V and had mp 264-265° (dec., from ethylene glycol). Found: N 26.1%. $C_{18}H_{20}N_6$. Calculated: N 26.2%.

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