CYCLIZATION OF 2-(1-METHYL-2-BUTENYL)ANILINE IN POLYPHOSPHORIC ACID

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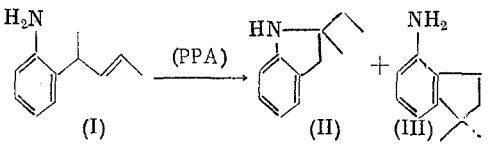
We have established that the cyclization of 2-(1-methyl-2-butenyl)aniline (I) in polyphosphoric acid (PPA) is accompanied by isomerization of the allyl fragment and leads to 2-methyl-2-ethylindoline (II). Together with it, we have also detected 4,4-dimethyl-1-aminoindan (III), a product of unusual cyclization of (I). The temperature and the ratio of (I) to polyphosphoric acid exert a significant effect on the composition of the reaction products:



Compound (I) (3 g) was heated in 10 g of polyphosphoric acid for 1.5-2 h at 150°C. Products (II) and (III) were isolated by chromatography on a column with Al_2O_3 (activity grade III), with benzene as the eluant. The yield of the indoline (II) was 75%, n_D^{20} 1.5465, R_f 0.86. IR spectrum (ν, cm^{-1}) 3380 (NH), 1610 (Ar). Proton NMR spectrum (CCl_4, TMS, δ, ppm): 0.83 t (3H, CH_3), 1.12 s (3H, CH_3), 1.41 m (2H, CH_2), 2.68 s (2H, CH_2), 3.26 s (1H, NH), 6.2-7.0 m (4H, Ar). Found, %: C 81.96; H 9.45; N 8.66. $C_{11}H_{15}N$. Calculated, %: C 81.99; H 9.32; N 8.70. The yield of the indan (III) was 15%, n_D^{20} 1.597, R_f 0.52. IR spectrum (ν, cm^{-1}): 3460, 3380 (NH_2), 1620 (Ar). Proton NMR spectrum (CCl_4, TMS, δ, ppm): 1.6 s (6H, $2CH_3$), 1.83 t (2H, CH_2), 2.53 t (2H, CH_2), 3.25 s (2H, NH_2), 6.6 m (3H, Ar). Found, %: C 81.92; H 9.37; N 8.60. $C_{11}H_{15}N$. Calculated, %: C 81.99; H 9.32; N 8.70.

By heating 1 g of the amine (I) in 10 g of polyphosphoric acid at 135°C for 2 h, we obtained 0.61 g (61%) of (III) and 0.2 g (20%) of (II).

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