LETTERS TO THE EDITOR

SYNTHESIS OF 2-[(2-METHYL-6-ETHYLANILINO)METHYL]-TETRAHYDROFURAN VIA RADICAL AMINOALKYLATION

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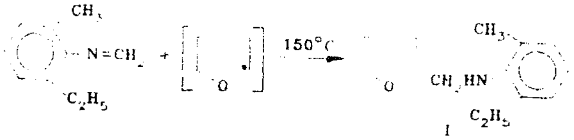
The introduction of an aminoalkyl function into the tetrahydrofuran ring under radical-reaction conditions remains virtually uninvestigated; at the same time, the advances in the photochemistry of imines attest to the possibility of the amination of cyclic ethers [1]. However, the interest in these reactions is due more to the urge to ascertain their mechanisms than to the possibility of their practical utilization because of the low yields obtained.

We have studied the homolytic aminoalkylation of tetrahydrofuran with N-methylene-2-methyl-6-ethylaniline, which is initiated by tert-butyl hydroperoxide and leads to 2-[(2-methyl-6-ethylanilino)methyl]tetrahydrofuran. The 2-tetrahydrofuranyl radicals were generated under standard conditions [2].



I

A mixture of 0.2 mole of tetrahydrofuran, 0.05 mole of the azomethine, and 0.03 mole of tert-butyl hydroperoxide was thermostatted in an ampul for 2 h at 150°C. After removal of the low-boiling compounds with a rotary evaporator, the residue was subjected to fractional distillation in vacuo. The conversion of the azomethine reached 40%. The yield of amine I was 90% based on the converted azomethine.

2-[(2-Methyl-6-ethylanilino)methyl]tetrahydrofuran (I) is a colorless liquid with bp 150°C (1 hPa), n_D^{20} 1.5595, and d_4^{20} 3932. IR spectrum: 1020, 1075, 1210, 1250, 1370, 1460, 1590, 3380 cm^{-1}. PMR spectrum ($CDCl_3$): 0.99 (3H, t, CH_3), 1.44-1.74 (4H, m, $2CH_2$), 2.06 (3H, s, CH_3), 2.48 (2H, q, CH_2), 2.66-2.86 (1H, m, CH), 3.40-3.88 (4H, m, $2CH_2$), 6.60-6.98 ppm (3H, m, H_{Ar}). The fragmentation of I by the action of electron impact is basically similar to the fragmentation of substituted cyclic ethers [3]. In addition to molecular-ion peaks (m/z 218, 13%) and $[M - CH_3]^+$ (17%) and $[M - C_2H_5]^+$ (14%) peaks in the mass spectrum, one observes a characteristic (for 2-substituted tetrahydrofurans) peak at 71 (42%), which is formed in the dissociation of the molecular ion at the exocyclic bond, as well as the aniline fragment at 134 (100%) that is characteristic for secondary amines.

LITERATURE CITED

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