A NEW REACTION OF METHYL TRIFLUOROPYRUVATE WITH ANILINE

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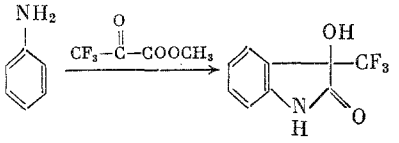
Saloutin et al. [1] have reported that methyl trifluoropyruvate reacts with aniline to form the N-alkylation product. We have found that carrying out this reaction in benzene at 120°C leads to 3-hydroxy-3-trifluoromethylbenzo[b]pyrrolidin-2-one (I).



(I)

The structure of pyrrolidone (I) was supported by ^{13}C NMR spectral data, mass spectral data, and elemental analysis. The ^{13}C NMR spectrum was taken on a Bruker WP-200SY spectrometer at 50.31 MHz. The chemical shifts were determined for acetone solutions relative to TMS as the internal standard.

3-Hydroxy-3-trifluoromethylbenzo[b]pyrrolidin-2-one (I). A solution of 1.40 g aniline and 3.12 g methyl trifluoropyruvate in 8 ml anhydrous benzene was heated in a glass ampul for 6 h at 120°C. The ampul was opened, and the crystals formed were filtered off and washed with hexane. The mother liquor was evaporated to yield additional product. The total yield of white crystalline (I), mp 215-217°C, was 2.0 g (61.44%), R_f 0.25 (3:1 CCl_4—acetone). ^{13}C—{^1H} NMR spectrum (δ, ppm): 76.01 (C^3, $^2J_{C-F}$ = 29.06 Hz), 111.32 (C^7), 123.45 (C^5), 124.60 (CF_3, $^1J_{C-F}$ = 285.80 Hz), 125.33 (C^9), 126.77 (C^6), 132.26 (C^4), 143.78 (C^8), 172.51 (C^2). Found: C 40.66; H 2.80; N 6.38; F 26.19%. Calculated for $C_9H_6NO_2F_3$: C 49.76; H 2.76; N 6.45; F 26.26%, M^+ 217.

LITERATURE CITED

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