SYNTHESIS OF ETHYL 2-OXO-2,3-DIHYDRO-1H-1,5-BENZODIAZEPINE-4-CARBOXYLATE

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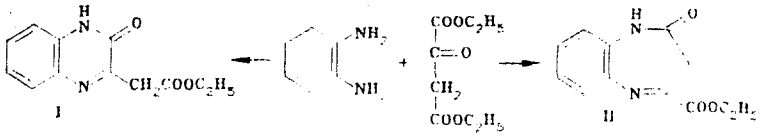
We have previously shown [1, 2] that 3-ethoxycarbonylmethyl-1H-quinoxal-2-one (I) is formed when o-phenylenediamine is heated with oxaloacetic ester in alcohol for 15-20 min.



We have established that ethyl 2-oxo-2,3-dihydro-1H-1,5-benzodiazepine-4-carboxylate (II), with mp 157-158°C (from benzene or dioxane), is formed in 70% yield when the starting substances are refluxed in o-xylene for 1 h. The IR spectrum of diazepinone II contain bands of NH stretching vibrations (3310-3350 cm^{-1}), a secondary amide band (1670 cm^{-1}), and an ester carbonyl band (1710 cm^{-1}). PMR spectrum: 1.35 (t, CH_3), 4.35 (q, CH_2), 3.55 (s, 3-H), 10.5 (broad s, NH), 6.8-7.4 ppm (m, H_{arom}). In contrast to the mass spectrum of quinoxaline I, the mass spectrum of diazepine II contains an [M – 42] peak with m/z 190, which is characteristic for the fragmentation of such systems [3]. Mass spectrum, m/z (I_{rel}, %): 232 (41) $[M]^+$, 190 (52) [M – CH_2CO], 159 (100) [M – $COOC_2H_5$], 131 (30) [159 – CO].

As we have shown, quinoxalone I is formed in better yield when the starting substances are stirred at room temperature; it has mp 216-218°C (from alcohol) and is formed in 96% yield. Mass spectrum, m/z (I_{rel}, %): 232 (99) $[M]^+$, 187 (46) [M – OC_2H_5], 186 (100) [M – C_2H_5OH], 158 (55) [186 – CO], 130 (42) [158 – CO].

The results of elementary analysis for C, H, and N were in agreement with the calculated values.

LITERATURE CITED

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