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UNUSUAL EXAMPLE OF THE FORMATION OF A 1-AZA-3,6-DIOXABICYCLO[3.2.1]OCTANE DERIVATIVE

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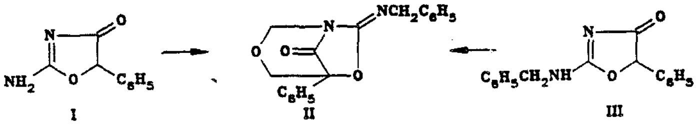
We have found that 2-amino-5-phenyl-4-oxazolinone (I) upon treatment with benzylamine and paraform does not undergo the expected aminomethylation reaction, but rather undergoes successive transamination [1], bishydroxymethylation, and dehydration reactions, to give 7-benzylimino-8-oxo-5-phenyl-1-aza-3,6-dioxabicyclo[3.2.1]octane (II). Compound II was also obtained by an independent synthesis from 2-benzylamino-5-phenyl-4-oxazolinone (III) and formalin



Compound II. A. A mixture of 10.6 g (0.06 mole) compound I, 9.6 g (0.09 mole) benzylamine, 7.7 g (0.25 mole) paraform, and 34.3 ml (0.60 mole) acetic acid in 180 ml methanol was refluxed for 2 h; the reaction mixture was then cooled to 20°C and neutralized with 20% aqueous potassium hydroxide. The resulting precipitate was crystallized from ethanol. Yield 2.0 g (11%), mp 190°C. IR spectrum (in KBr): 1825 (C=O), 1740 cm^{-1} (C=N). PMR spectrum (CCl_4): 7.00-7.42 (10H, m, C_6H_5), 4.03-4.70 (2H, m, 2-CH_2), 2.90-4.03 ppm (4H, m, 4-CH_2, CH_2Ph).

B. A mixture of 0.85 g (0.03 mole) compound III, 0.95 ml (0.013 mole) formalin, and 1.5 ml (0.027 mole) acetic acid in 4.5 ml methanol was refluxed for 0.5 h and worked up to give compound II, as described above. Yield 0.05 g (5%), mp 188°C. The results of elemental analysis were consistent with the calculated values.

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