N-ALLENYLATION OF PYRROLES USING 1,2,3-TRICHLOROPROPANE

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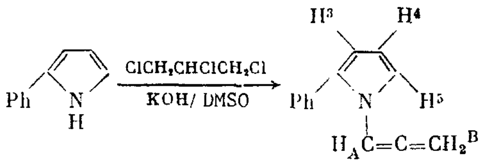
We have found that 1,2,3-trichloropropane is an efficient allenylating agent for pyrroles in the KOH/DMSO superbase system. Thus, 1-propadienyl-2-phenylpyrrole (I) was obtained in 62% yield directly from 2-phenylpyrrole. The double dehydrochlorination, nucleophilic substitution of the chlorine atom by the pyrrolate anion, and prototropic isomerization of intermediate 1-(2-propynyl)-2-phenylpyrrole to an allene were carried out in a single preparative step with almost 100% selectivity.



(I)

A sample of 5.53 g 1,2,3-trichloropropane was added over 10 min to a mixture of 3.56 g 2-phenylpyrrole, 11.2 g KOH (subjected to prior roasting at 500°C for 4 h), and 50 ml dry DMSO (containing 0.2% H_2O). Heat was evolved and the temperature rose to 40°C. The mixture was then heated for an additional 20 min at 40°C and 100 ml water was added. The reaction mixture was extracted with ether. The ethereal extracts were washed with water and dried over $MgSO_4$. Ether was evaporated and chromatography of the residue on alumina with hexane as the eluent gave 2.8 g of previously reported allene (I), n_D^{20} 1.6370. PMR spectrum (δ, ppm): 6.94 t (1H, H_A), 5.43 d (2H, H_B), $^4J_{H_AH_B}$ 6.6 Hz, 6.94 t (1H, H^5), 6.26 d (2H, H^3, H^4), 7.36 m (5H, Ph). IR spectrum (ν, cm^{-1}): 1950 (C=C=C), 1604, 1500 (Ph), 1475, 1380 (pyrrole ring), 1314 (C–N). The elemental analysis corresponded to the proposed chemical formula.

In light of the availability of 1,2,3-trichloropropane, which is produced in large amounts as a by-product in the production of epichlorohydrin, this simple and efficient method for allenylation under mild conditions holds promise for use in the synthesis of various heterocyclic compounds with a labile hydrogen atom.

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