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AMINATION OF PYRIDONES BY HEXAMETHYL TRIAMIDOPHOSPHATE

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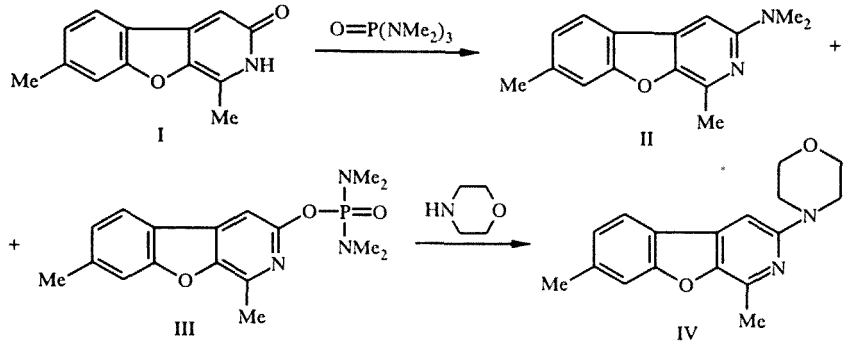
In a study of the amination of 1,7-dimethyl-3(2H)-benzo[b]furo[2,3-c]pyridone (I) by hexamethyl triamidophosphate according to a reported procedure [1-3], we found that O-(1,7-dimethylbenzo[b]furo[2,3-c]pyrid-3-yl)tetramethyl diamidophosphate (III) is formed along with 1,7-dimethyl-3-dimethylaminobenzo[b]furo[2,3-c]pyridone (II). Product III is the major product in the case of heating for only 2-3 h.



Further heating of the mixture leads to an increase in amino derivative II. In our opinion, III is an intermediate in amination reactions using hexamethyl triamidophosphate. Indeed, heating amidophosphate III with excess secondary amine such as morpholine leads to the corresponding 3-morpholino derivative IV. Previous attempts to isolate intermediates in such reactions have been unsuccessful [4].

O-(1,7-Dimethylbenzo[b]furo[2,3-c]pyrid-3-yl)tetramethyl Diamidophosphate (III), mp 125-126°C. PMR spectrum (in pyridine-d_5, TMS): 2.38 (2H, s, 7-CH_3), 2.68 (2H, s, 1-CH_3), 2.81 (6H, s, $N(CH_3)_2$), 2.86 (6H, s, $N(CH_3)_2$), 7.14 (1H, d, J = 8 Hz, 6-H), 7.44 (1H, s, 4-H), 7.78 (2H, s, 8-H), 7.84 (1H, d, J = 8 Hz, 5-H). Thin-layer chromatography on Silufol UV-254, R_f 0.31 (3:1 ethyl acetate—benzene). Yield 58%.

The elemental analysis data corresponded to the calculated values.

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