SYNTHESIS OF 2-ETHYL-1,3-DIBUTYL-4,5-DIMETHYL-1,3,2-DIAZAPHOSPHOLENE

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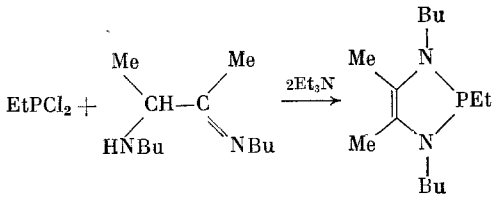
In our previous work [1], we showed that the reaction of 2-ethyl-2-oxo-1,3-dibutyl-4,5-dimethyl-1,3,2-diazaphospholene with trichlorosilane in the presence of triethylamine gives a good yield of 2-ethyl-1,3-di-butyl-4,5-dimethyl-1,3,2-diazaphospholene. In the present communication, we present two new methods for the preparation of this diazaphospholene. The first method entails the addition of a 1:2 mixture of 2-butylamino-3-butyliminobutane and triethylamine to ethyldichlorophosphine in absolute ether at 5-10°C (the mole ratio of the iminobutane and phosphine was 1:1). After standing overnight, the hydrochloride salt o triethylamine was filtered off. The solvent was removed. The residue was distilled to give a 56% yield of 2-ethyl-1,3-dibutyl-4,5-dimethyl-1,3,2-diazaphospholene, bp 130°C (10 mm), n_D^{20} 1.4820, $\delta^{31}P$ +112 ppm [1].



In the second method, this diazaphospholene is obtained as a result of the reaction of ethyldichlorophosphine with the sodium derivative of N, N'-dibutyl-2,3-butanediimine in THF. The product yield was 43%, bp 135-136°C (13 mm), n_D^{20} 1.4851, $\delta^{31}P$ +110 ppm.

LITERATURE CITED

1. A. M. Kibardin, Yu. B. Mikhailov, T. V. Gryaznova, and A. D. Pudovik, Izv. Akad. Nauk SSSR, Ser. Khim., 960 (1986).

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