

LETTERS TO THE EDITOR

PREPARATION OF 1,4-DIPHENYLPIPERAZINE

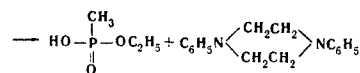
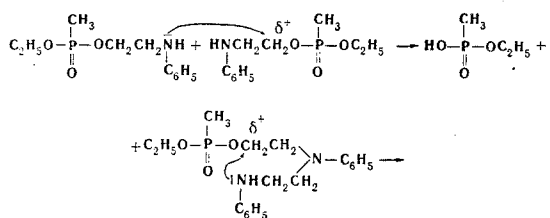
O. P. Kor'yakov, P. M. Zavlin, and V. V. Razumovskii

Khimiya Geterotsiklicheskikh Soedinenii, Vol. 4, No. 6, p. 1131, 1968

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When 17.3 g of ethyl β -phenylaminoethyl methylphosphonate (n_D^{20} 1.5491, structure established on the basis of the results of microanalysis and infrared spectroscopy) isolated from the product of the interaction of ethyl methylphosphonochloridate and β -phenylaminoethanol, was subjected to thermal decomposition, in the absence of a solvent, in a current of argon at 200° C for 1 hr, 8.1 g (92%) of 1,4-diphenylpiperazine was formed. After two recrystallizations from *o*-xylene, it had mp 164–164.5° C (according to the literature [1–3], mp 164–165° C).

Formation of 1,4-diphenylpiperazine in the case under consideration is apparently the result of successive processes of inter- and intramolecular alkylation of the amino group of the ethyl β -phenylaminoethyl methylphosphonate, taking place in the following way:



In addition to 1,4-diphenylpiperazine, a mixture of polyphosphonates, apparently products of the polycondensation of ethyl methylphosphonate, is formed.

REFERENCES

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Bonch-Bruевич Leningrad Electro-technical Institute of Communication

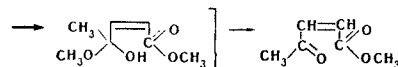
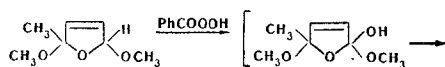
PREPARATION OF METHYL β -ACETYLACRYLATE

R. I. Kruglikova, L. N. Kralinina, and T. V. Boyarinova

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When 2,5-dimethoxy-2,5-dihydroxylane (I) was oxidized with perbenzoic acid, instead of the expected 3,4-epoxy-2,5-dimethoxy-tetrahydroxylane, methyl β -acetylacrylate (II) was formed. This compound has been obtained previously in other ways [1–4]. It is interesting that II possesses bacteriostatic activity against *Escherichia coli*, *Mycobacterium tuberculosis* and *Pneumococcus* [4]. The constants of the II that we obtained and of its dinitrophenylhydrazone agreed with those given in the literature. Furthermore, the structure of II was confirmed by its IR and NMR spectra. The IR spectrum had strong absorption bands at 1725 and 1680 cm^{-1} and a less intense band at 1645 cm^{-1} . The NMR spectra had two singlets with chemical shifts of 2.24 ppm ($\text{CH}_3\text{C}=\text{O}$) and 3.76 ppm ($\text{CH}_3\text{-O-C}=\text{O}$) and a doublet at 6.67 and 6.77 ppm ($-\text{CH}=\text{CH}-\text{C}=\text{O}$) with intensities of 3:3:2.



Obviously, in view of the fact that the multiple bond in the 2,5-dialkoxy-2,5-dihydrofurans is not oxidized under these conditions [5], the perbenzoic acid oxidizes the acetal group and the reaction takes place in the manner shown above. We consider this course of the reaction the most probable, since there is information on the oxidation with peracetic acid of α, β -saturated acetals to the corresponding esters [6].

A similar conversion was observed in the oxidation of 2,5-dimethoxy-2,5-dihydrofurfuryl alcohol with performic acid (β -benzoyloxydehydrolevulinic acid was obtained) [7].

Compound II has been obtained by the oxidation of I with a twofold excess of perbenzoic acid in chloroform solution, yield 58%, mp 61–62° C (from *n*-hexane); 2,4-dinitrophenylhydrazone, mp 200–202° C.