A SIMPLE VARIANT OF THE MICHAELIS—BECKER PHASE TRANSFER REACTION

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A convenient variant of the Michaelis—Becker reaction is the alkylation of $(RO)_2PHO$ in the presence of potassium carbonate and a phase transfer catalyst [1]. However, the use of this method involves a number of difficulties related to the synthesis of compounds with good solubility in water and in those cases when the catalyst cannot be simply removed from the reaction products.

We have developed a sample method for the alkylation of $(RO)_2PHO$ and analogous compounds in the presence of potassium carbonate without a phase transfer catalyst. This method is especially convenient in the case of $(MeO)_2PHO$. No information on the alkylation of this compound is available. A sample of 41.9 g (295 mmoles) MeI was added dropwise with rapid stirring to a mixture of 25.0 g (227 mmoles) $(MeO)_2PHO$ and 62.6 g (454 mmoles) dry, ground K_2CO_3 at 20-25°C. The mixture was stirred for 4 h at 25°C and diluted with 25 ml $CHCl_3$ and 1 ml water. The residue was filtered off. The filtrate was evaporated in vacuum and the residue was distilled. The yield of O,O-dimethyl methylphosphonate was 19.4 g (70%), bp 58-60°C (10 mm). ^{31}P NMR spectrum: 33.7 ppm [2]. Analogous procedures gave O,O-diethyl methylphosphonate in 88% yield from $(EtO)_2PHO$ and MeI upon carrying out the reaction at 30°C over 8 h, bp 69-69.5°C (6 mm), ^{31}P NMR spectrum: 29.9 ppm [2], and also diphenyl(ethoxycarbonylmethyl)phosphine oxide from Ph_2PHO and $ClCH_2CO_2Et$ in 81% yield upon carrying out the reaction at 55-60°C over 1 h, mp 75.5-76.5°C (from ethyl acetate), ^{31}P NMR spectrum in ethanol: 27.2 ppm [3].

LITERATURE CITED

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