

LETTERS
TO THE EDITOR

Reaction of Spiro[2.2]pentane with Phosphorus Pentachloride

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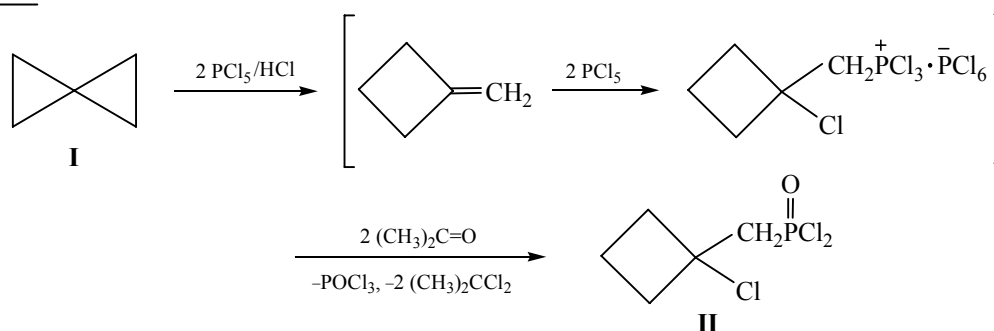
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Due to the high strain energy spiranes containing small cycles are interesting objects for phosphorylation with phosphorus compounds of various coordination. It is known that one of the available mild phosphorylating agents is phosphorus pentachloride possessing electrophilic character [1]. Therefore, its reaction with such spiranes may occur through an opening of a three- or four-membered rings to

produce new types of phosphorylated cyclic hydrocarbons.

We showed that the interaction of spiro[2.2]pentane **I** with phosphorus pentachloride under standard conditions [2] (benzene, 0–20°C, quenching reagent acetone) resulted in 1-chlorocyclobutyl-methylphosphonic acid dichloride **II**.



Apparently, first the three-membered ring of spiro [2.2]pentane opened to form methylenecyclobutane, which further is phosphorylated by the known scheme [3].

The structure of compound **II** was confirmed by the IR and ^1H NMR spectroscopy. The IR spectrum contains absorption bands of stretching vibrations of the bonds P=O (1275 cm^{-1}), C–Cl (755 cm^{-1}) and P–Cl ($580, 540\text{ cm}^{-1}$). The analysis of the ^1H NMR spectrum confirms the retention of the cyclobutane ring. The signals of methylene protons in 2,4-positions of the ring appear as multiplets at 2.73 and 2.86 ppm, and in position 3, as multiplets at 2.15 and 2.39 ppm, respectively. The methylene group at the phosphorus atom resonates as a characteristic doublet at 3.57 ppm ($^2J_{\text{HP}}$ 13.8 Hz).

Dichloride **II** is a colorless liquid that crystallized at 20–21°C, soluble in common organic solvents.

The IR spectrum was recorded on a Fourier transform infrared spectrometer FSM-1202 in the range of 400–4000 cm^{-1} from KBr pellets. The ^1H NMR spectrum was taken on a spectrometer Bruker WM-250 (250 Hz) relative to internal dimethyl sulfoxide.

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