REACTION OF SULFURYL CHLORIDE WITH P-TRIMETHYLSILYL-C-TERT-BUTYL-C-TRIMETHYLSILYLOXYMETHYLENEPHOSPHINE

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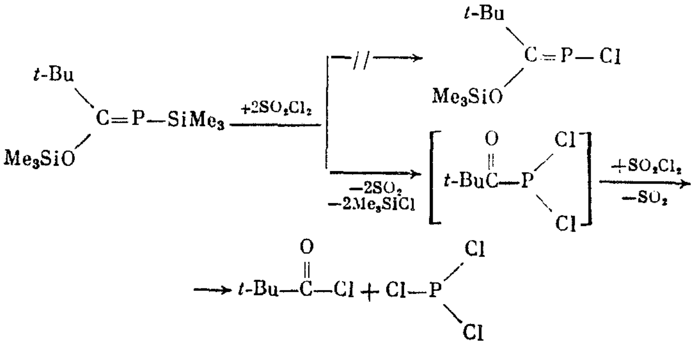
Appel and Barth [1] have shown that P-silylphosphaalkenes may be converted by the action of hexachloroethane to P-chlorophosphaalkenes although chlorination at the P=P bond does not proceed.

We have shown that chlorination occurs both at the P-Si and P=C bond when SO_2Cl_2 is used for this purpose. Complete cleavage of the P=C bond occurs in this case:



It proved impossible to stop the chlorination at the step of the P-chlorophosphaalkene by varying the mole ratios of the starting reagents.

A sample of 0.88 g (0.034 mole) P-trimethylsilyl-C-tert-butyl-C-trimethylsilyloxymethylenephosphine was added dropwise to 1.42 g (0.0105 mole) sulfuryl chloride. After no further SO_2 was evolved, the reaction mixture was distilled, collecting PCl_3 at 73-74°C. The yield of PCl_3 was 0.20 g (43%). ^{31}P NMR spectrum: 219 ppm [2]. Distillation also gave 0.33 g (81%) pivaloyl chloride, bp 105-106°C, n_D^{20} 1.4120 [3].

LITERATURE CITED

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2. É. E. Nifant'ev and L. K. Vasyanina, ^{31}P NMR Spectroscopy [in Russian], Moscow State Pedagogical Institute, Moscow (1986), p. 34.
3. Aldrich Catalog of Fine Chemicals (1990-1991), p. 1295.

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