Russian Journal of General Chemistry, Vol. 73, No. 6, 2003, p. 993. Translated from Zhurnal Obshchei Khimii, Vol. 73, No. 6, 2003, p. 1050.
Original Russian Text Copyright © 2003 by M. Pudovik, Terent'eva, A. Pudovik.

LETTERS TO THE EDITOR

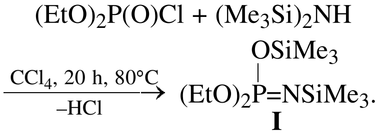
Synthesis of Diethyl Trimethylsilyl (Trimethylsilylimido)phosphate

M. A. Pudovik, S. A. Terent'eva, and A. N. Pudovik

Arbuzov Institute of Organic and Physical Chemitry,
Kazan Research Center, Russian Academy of Sciences, Kazan, Tatarstan, Russia

Received May 28, 2002

At present two methods of synthesis of dialkyl trimethylsilyl (trimethylsilylimido)phosphates have been described: exhaustive silylation of dialkyl imidophosphate with chlorotrimethylsilane in the presence of bases and the reaction of phosphorus acid chlorides with sodium or lithium bis(trimethylsilyl)amides [1, 2]. According to published data, the reaction of dialkyl(aryl) chlorophosphates with hexamethyldi silazane is accompanied by elimination of trimethylchlorosilane and stops at the stage of formation of dialkyl trimethylsilyl imidophosphates [3, 4]. We found that diethyl trimethylsilyl (trimethylsilylimido)phosphate (**I**) can be prepared by prolonged heating of equimolar amounts of diethyl chlorophosphate and hexamethyldisilazane in carbon tetrachloride. This method is simple in operation and permits one to vary the type of alkyl substituents in the final product.



$$(EtO)_2P(O)Cl + (Me_3Si)_2NH \xrightarrow[-HCl]{CCl_4,\ 20\ h,\ 80°C} (EtO)_2\overset{\overset{\textstyle OSiMe_3}{|}}{P}{=}NSiMe_3.$$

I

Diethyl trimethylsilyl (trimethylsilylimido)phosphate (I). A solution of 7.5 g of diethyl chlorophosphate and 7 g of hexamethyldisilazane in 50 ml of carbon tetrachloride was refluxed for 20 h. The solvent was removed, and the residue was distilled in a vacuum to obtain 5.8 g (45%) of compound **I**, bp 50–53°C (0.06 mm), n_D^{20} 1.4182 [3]. ^{31}P NMR spectrum: δ_P –12.28 ppm. Found, %: P 9.94; Si 18.85. $C_{10}H_{28}NOPSi$. Calculated, %: P 10.43; Si 18.83.

The ^{31}P NMR spectrum was recorded on a Bruker MSL-400 spectrometer (166.93 MHz) against external 85% phosphoric acid.

ACKNOWLEDGMENTS

The work was financially supported by the Russian Foundation for Basic Research (project no. 00-03-32837).

REFERENCES

1. Meyer, M. and Heyde, B., *Tetrahedron Lett.,* 1969, no. 18, p. 1425.
2. Wilburn, J.C., *Inorg. Chem.,* 1978, vol. 17, p. 1880.
3. Becke-Goering, M. and Wunsch, G., *Chem. Ber.,* 1960, vol. 93, p. 326.
4. Becke-Coering, M., *Angew. Chem.,* 1960, vol. 72, p. 463.

1070-3632/03/7306-0993 $25.00 © 2003 MAIK "Nauka/Interperiodica"