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LETTERS
TO THE EDITOR

S-Trialkylstannylbis(*N*,*N*-diethylamido)dithiophosphates

I. S. Nizamov, E. S. Batyeva, I. D. Nizamov, and V. A. Al'fonsov

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

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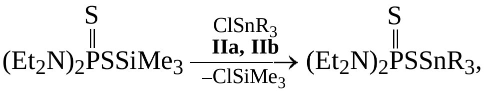
S-Trialkylstannyl derivatives of diamidodithiophosphoric acids up to now were unknown. These compounds could not be obtained by the reaction of tetraphosphorodecasulfide with trialkyl-*N*,*N*-diaminostannanes according to the previously developed procedures [1, 2] because of the unavailability of aminostannanes [3]. We offer a convenient method for preparing of *S*-trialkylstannyldiamidodithiophosphates by the reaction of interchange of trimethylsilyl group of *S*-silyldiamidodithiophosphates with trialkylstannyl one under the action of trialkylhalostannanes. It is found that *S*-trimethylsilylbis(*N*,*N*-diethylamido)dithiophosphate **I**, prepared by the reaction of tetraphosphorodecasulfide with trimethyl-*N*,*N*-dialkylaminosilanes [1, 2], reacts with trialkylchlorostannanes **IIa** and **IIb** at 20°C for 5 days in benzene solution or without a solvent to give *S*-trialkylstannylbis(*N*,*N*-diethylamido)dithiophosphates **IIIa** and **IIIb**, and trimethychlorosilane **IV**.



$$\underset{\textbf{I}}{(Et_2N)_2\overset{\overset{S}{\|}}{P}SSiMe_3} \xrightarrow[-ClSiMe_3\ \textbf{IV}]{ClSnR_3\ \textbf{IIa, IIb}} \underset{\textbf{IIIa, IIIb}}{(Et_2N)_2\overset{\overset{S}{\|}}{P}SSnR_3},$$

R = Me (**a**), Bu (**b**).

Compounds **IIIa** and **IIIb** are the representatives of the new type of organophosphorus compounds containing $N_2P(S)SSn$ fragment. Substitution of the silicon atom by the tin one in diamidodithiophosphoric compounds leads to the alteration in the chemical shift of phosphorus in ^{31}P NMR spectrum from δ_P 83.3 ppm (in C_6H_6) for *S*-silyldiamidodithiophosphate **I** (published data [2]: 83.6 ppm in CH_2Cl_2) to δ_P 86.8 ppm and 87.7 ppm respectively. In electron impact mass spectra of compounds **IIIa** and **IIIb** the peaks at *m/z* 403 (3%) and 529 (1%) corresponding to their molecular ions are present.

***S*-Trimethylstannylbis(*N*,*N*'-diethylamido)dithiophosphate (IIIa).** To a solution of 4.1 g of *S*-silyldiamidodithiophosphate **I** in 5 ml of anhydrous benzene 2.6 g of trimethylchlorostannane **IIa** was added dropwise with stirring at 20°C under argon. Resulting mixture was stirred for 5 h at 20°C, kept for ~12 h and filtered. Obtained filtrate was kept in a vacuum (0.5 and then 0.07 mm) for 1.5 h at 40°C. The volatile fractions were collected in a trap cooled with liquid nitrogen. Molecular film distillation of the residue gave 2.2 g (42%) of compound **IIIa** at the thermoelement temperature 120–125°C (0.03 mm), n_D^{20} 1.5500. IR spectrum, ν, cm^{-1}: 2970 v.s, 2930 s, 2912 sh., 2873 m [ν(CH_3 as,s; CH_2 as,s)], 1460 m [δ(CH_3 as)], 1030 v.s [γ(C–N–C as)], 790 v.s [ρ(Sn–C)], 621 m.b [ν(P=S)], 535 m, 509 w [ν(P–S), γ(Sn–C)]. ^1H NMR spectrum, δ, ppm (*J*, Hz): 0.67 s [9H, $(CH_3)_3Sn$], 1.18 t [12H, $(CH_3CH_2)_2NP$, $^3J_{HH}$ 6.5, $^3J_{PH}$ 13.0]. Found, %: P 8.18; S 15.37; Sn 29.42. $C_{11}H_{29}N_2PS_2Sn$. Calculated, %: P 7.69; S 15.87; Sn 29.46. *M* 402.9.

Distillation of the volatile fraction gave 0.9 g (64%) of chlorosilane **IV**, bp 57°C, n_D^{20} 1.3890 (published data [4]: bp 57.5°C, n_D^{20} 1.3885).

***S*-Tributylstannylbis(*N*,*N*'-diethylamido)dithiophosphate (IIIb).** This compound was obtained analogously (in the absence of solvent), yield 60%, isolation by molecular film distillation at the thermoelement temperature 135–140°C (0.3 mm) , n_D^{20} 1,5290. IR spectrum, ν, cm^{-1}: 2960 v.s, 2926 v.s, 2872 s, 2855 s [ν(CH_3 as,s; CH_2 as, s)], 1460 m [δ(CH_3 as)], 1029 v.s [γ(C–N^3C as)], 792 m [ρ(Sn–C)], 623 m [ν(P=S)], 525 m.b [ν(P–S), γ(Sn–C)]. ^1H NMR spectrum, δ, ppm (*J*, Hz): 0.84–1.97 m [27H, $(C_4H_9)_3Sn$ + $(CH_3CH_2)_2NP$], 3.21 d.q [8H, $(CH_3CH_2)_2NP$, $^3J_{HH}$ 6.5, $^3J_{PH}$ 13.0]. Found, %: P 6.05; S 12.02; Sn 22.17. $C_{20}H_{47}N_2PS_2Sn$. Calculated, %: P 5.85; S 12.09; Sn 22.43. *M* 529.1.

IR spectra were recorded on an UR-20 spectro-

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meter. ^1H NMR spectra were measured on a Varian T-60 spectrometer (60 MHz) in CCl_4 solutions against internal CH_2Cl_2. ^{31}P NMR spectra were obtained on a Bruker MSL-400 spectrometer (162 MHz) against 85% phosphoric acid in C_6H_6. Mass spectra were measured on a Hitachi M-80B mass spectrometer.

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