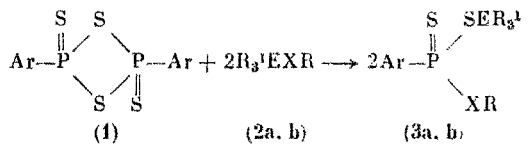
# S-TRIALKYLSILYL- AND S-TRIALKYL-STANNYL-4-METHOXYPHENYLPHOSPHONO-TRITHIO- AND DITHIOATES

I. S. Nizamov, V. A. Kuznetsov, É. S. Batyeva,
and V. A. Al'fonsov

UDC 542.91:547.1'128'118

We have found that 2,4-bis(4-methoxyphenyl)-2,4-dithioxo-1,3,2λ$^5$,4λ$^5$-dithiadiphosphetane (Lawesson's reagent) (**1**) reacts with isobutylthiotrimethylsilane (**2a**) and methoxytributylstannane (**2b**) at 20°C over 11 h (for **2a**) or 15°C over 1 h (for **2b**) to give S-isobutyl-S'-trimethylsilyl-4-methoxyphenylphosphonotrithioate (**3a**) and O-methyl-S-tributylstannyl-4-methoxyphenylphosphonodithioate (**3b**), respectively.



(**1**) (**2a, b**) (**3a, b**)

Ar=4-MeOC$_6$H$_4$; R=Bu-*i* (**a**), Me (**b**); R'=Me (**a**), Bu (**b**);
X=S (**a**), O (**b**); E=Si (**a**), Sn (**b**).

**S-Isobutyl-S'-trimethylsilyl-4-methoxyphenylphosphonotrithioate (3a)** was obtained in 76% yield. The temperature of the heating element in the molecular film distillation was 175-180°C (0.02 mm), $d_4^{20}$ 1.1202, $n_D^{20}$ 1.5979. Found: C, 46.32; H, 6.60; P, 8.98; S, 26.72; Si, 7.25%. Calculated for $C_{14}H_{25}OPS_3Si$: C, 46.14; H, 6.93; P, 8.51; S, 26.345; Si, 7.68%. IR spectrum ($\nu$, cm$^{-1}$): 3070, 3010 (:C—H Ar) 1595, 1500, 1465 ( C⋯ CAr ), 1387, 1370 $\delta$ ($Me_2C$ gem), 1260 [$CH_3$(Si)], 851 $\rho$ [$CH_3$(Si)], 690 (P=S), 540, 510 (S—Si, P—S, $PS_2$). PMR spectrum at 60 MHz in $CCl_4$ ($\delta$, ppm, $J$, Hz): 0.48 s (9H, C$\underline{H}_3$Si), 1.03 d (6H, C$\underline{H}_3$CHCH$_2$, $^3J_{H\text{-}H}$ = 7.0), 1.57-2.93 m ($CH_3C\underline{H}CH_2$, $CH_3CH\underline{CH_2}$), 3.87 s (3H, $CH_3O$), 6.86 d.d (2H, 3-$\underline{H}_2C_6H_2$, $^3J_{H\text{-}H}$ = 9.0, $^4J_{P\text{-}H}$ = 4.0), 7.96 d.d (2H, 2-$H_2C_6\underline{H}_2$, $^3J_{H\text{-}H}$ = 9.0, $^3J_{P\text{-}H}$ = 15.0). $^{31}$P NMR spectrum at 10.2 MHz relative to 85% $H_3PO_4$ ($\delta$, ppm): 71. Chemical ionization mass spectrum (*i*-$C_4H_{10}$, 100 eV), $m/z$ ($I_{rel}$): 276 [M + H — SBu-*i*]$^+$ (30), 203 [M + H — SBu-*i* — SSiMe$_3$]$^+$ (40).

**O-Methyl-S-tributylstannyl-4-methoxyphenylphosphonodithioate (3b)** was obtained in 74% yield. The temperature of the heating element in the molecular film distillation apparatus was 170-190°C (0.02 mm), $d_4^{20}$ 1.2297, $n_D^{20}$ 1.5233. Found: C, 45.44; H, 7.02; P, 5.99; S, 11.96; Sn, 22.95%. Calculated for $C_{20}H_{37}O_2PS_2Sn$: C, 45.89; H, 7.15; P, 5.92; S, 12.22; Sn, 22.70%. IR spectrum ($\nu$, cm$^{-1}$): 3010 (:C—H Ar), 1598, 1500, 1465 ( C⋯ CAr ), 1035 [PO—(C)], 788 $\rho$ (Sn—C), 686 (P=S), 538, 527 (Sn—C, P—S). PMR spectrum at 60 MHz in $CCl_4$ ($\delta$, ppm, $J$, Hz): 0.82-1.67 m (27H, $C_4\underline{H}_9$Sn), 3.70 d (3H, $CH_3OP$, $^3J_{P\text{-}H}$ = 15.0), 3.77 s (3H, C$\underline{H}_3$OC$_6$H$_4$), 6.85 d.d (2H, 3-$\underline{H}_2C_6H_2$, $^3J_{H\text{-}H}$ = 9.0, $^4J_{P\text{-}H}$ = 4.0), 7.85 d.d (2H, 2-$H_2C_6\underline{H}_2$, $^3J_{H\text{-}H}$ = 9.0, $^3J_{P\text{-}H}$ = 15.0). $^{31}$P NMR spectrum at 10.2 MHz relative to 85% $H_3PO_4$ ($\delta$, ppm): 100. Chemical ionization mass spectrum (isobutane, 100 eV), $m/z$ ($I_{rel}$): 525 [M + H]$^+$ (90), 492 [M + H — S]$^+$ (100), 467 [M + H — Bu]$^+$ (50), 435 [M + H — Bu — S]$^+$ (25), 291 [M + H —$MeOC_6H_4PS_2OMe$]$^+$ (90), 203 [M + H — $SnBu_3$ — OMe]$^+$ (50).

A. E. Arbuzov Institute of Organic and Physical Chemistry, Kazan Science Center, Russian Academy of Sciences, 420083 Kazan'. Translated from Izvestiya Akademii Nauk, Seriya Khimicheskaya, No. 10, pp. 2457-2458, October, 1992. Original article submitted June 8, 1992.

 1063-5211/92/4110-1936$12.50 