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LETTERS TO THE EDITOR

Reaction of Lawesson's Reagent with Dibutylbis(ethylthio)stannane

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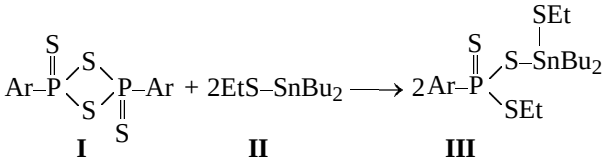
We previously studied the reactivity of 1,3,2λ^5,4λ^5-dithiadiphosphetane 2,4-disulfides toward geminal bis(alkylthio) derivatives of Group IV elements using the reaction with thioacetals as an example [1]. Reactions of bis(alkylthio)stannanes containing two S–Sn bonds with 1,3,2λ^5,4λ^5-dithiadiphosphetane 2,4-disulfides were not examined.

We have found that 2,4-bis(4-methoxyphenyl)-1,3,2λ^5,4λ^5-dithiadiphosphetane 2,4-disulfide (**I**, Lawesson's reagent) reacts with dibutylbis(ethylthio)-stannane (**II**) (10°C, 4 h), yielding dibutyl(ethylthio)-stannyl ethyl 4-methoxyphenylphosphonotrithioate (**III**). Thus, the reaction occurs under mild conditions and involves only one Sn–S bond.



I **II** **III**

Ar = 4-$MeOC_6H_4$.

Dibutyl(ethylthio)stannyl ethyl 4-methoxyphenylphosphonotrithioate (III). To a solution of 2.0 g of dithiostannane **II** in 10 ml of dry benzene, stirred at 10°C under dry argon, we added in portions 1.1 g of Lawesson's reagent (**I**). The mixture was stirred for 4 h at 10°C and filtered. The filtrate was evacuated under a residual presure of 0.5 and 0.04 mm for 2 h at 40°C. The residue was subjected to film distillation to isolate 1.5 g (50%) of product **III**, thermoelement temperature 150°C (0.06 mm), n_D^{20} 1.5728. IR spectrum, ν, cm^{-1}: 3070 w, 3030 sh (C–H_{arom}); 2963 s, 2930 s, 2875 m, 2860 m (C–H in CH_3 and CH_2, sym. and asym.); 1590 s, 1500 s, 1460 m (C=C_{arom}), 1260 s (ω, τCH_2); 675 m, very broad (P=S); 623 m (C–C); 545 m, br (Sn–S); 515 m (P–S). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.73–2.05 m (27H, C$H_3$$CH_2$S, $CH_3CH_2CH_2CH_2$Sn), 2.33–3.07 m (4H, $CH_3$$^{-1}H_2$S), 3.83 s (3H, CH_3O), 6.87 d.d (2H, *m*-H, $^3J_{HH}$ = 9.0, $^4J_{PH}$ = 3.0), 8.06 d.d (2H, *o*-H, $^3J_{HH}$ = 9.0, $^3J_{PH}$ = 15.0). ^{31}P NMR spectrum: δ_P 77.0 ppm. Found, %: P 5.56; Sn 21.31. $C_{19}H_{35}OPS_4Sn$. Calculated, %: P 5.31; Sn 21.08.

The IR spectrum was taken on a UR-20 spectrometer. The ^{1}H spectrum was recorded on a Varian T-60 instrument (60 MHz) in $CDCl_3$ containing HMDS as internal reference. The ^{31}P NMR spectrum was obtained on a Bruker MSL-400 spectrometer at 162 MHz using benzene as solvent and 85% H_3PO_4 as reference.

ACKNOWLEDGMENTS

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