Preparative synthesis of trialkylstannanes

*Yu. A. Turkin, S. Kh. Khangazheev, R. G. Mirskov, R. G. Voronkov, and V. I. Rakhlin**

Irkutsk Institute of Organic Chemistry, Siberian Branch of the Russian Academy of Sciences, 1 ul. Favorskogo, 664033 Irkutsk, Russian Federation. Fax: +7 (395 2) 466 434

Reactions of 1,1,3,3-tetramethyldisiloxane with compounds containing an Sn—O bond were studied. Trialkylstannanes are the major products of the reactions with trialkylalkoxystannanes and -stannoxanes.

Key words: 1,1,3,3-tetramethyldisiloxane, trialkylstannane, reduction, trialkylalkoxystannane.

Trialkylstannanes (**1**) are widely applied in fine organic synthesis as highly selective reducing agents.[1] The most commonly used methods for the synthesis of these compounds are based on reduction of trialkylhalostannanes with lithium tetrahydroaluminate[2] or diethylaluminum hydride.[3]

We have developed a convenient technique for preparing compound **1**, based on the reaction of compounds containing Sn—O bonds with 1,1,3,3-tetramethyldisiloxane (**2**), which acts as a reducing agent:

$$\underset{\mathbf{3}}{R_3SnOR'} + \underset{\mathbf{2}}{HMe_2SiOSiMe_2H} \longrightarrow R_3SnH + \underset{\mathbf{1}}{R'OMe_2SiOSiMe_2H}$$

R = Me, Bu; R' = Me, R_3Sn

In the case of hexaalkyldistannoxanes, the reaction affords two molecules of **1** and a mixture of oligomeric dimethylsiloxanes. The yields of trialkylstannanes are 89—94 %, their purity is no less than 99.5 %, according to GLC. An attempt to react dibutyltin oxide with compound **2** was unsuccessful.

Experimental

GLC analysis was carried out on a Tsvet-500 chromatograph using a heat-conductivity detector, helium as the carrier gas, 3 m×4 mm glass columns, and Inerton-super sorbent with a grain size of 0.2—0.25 mm, impregnated with 10 % PMS-1000.

Reaction of triethylmethoxystannane with 1,1,3,3-tetramethyldisiloxane. 1,1,3,3-Tetramethyldisiloxane (13.45 g, 0.1 mol) was placed in a round-bottom three-necked flask equipped with a reflux condenser, a dropping funnel, and a mechanical stirrer, and triethylmethoxystannane (47.4 g, 0.2 mol) was added dropwise with intense stirring. After the addition, the reaction mixture was stirred for 2 h and distilled *in vacuo* to give 36.8 g (89 %) of triethylstannane, b.p. 69—70 °C (47 Torr), n_D^{20} 1.4725 (*cf.* Ref. 3: b.p. 148—150 °C (76 Torr), n_D^{20} 1.4709).

Reaction of hexapropyldistannoxane with 1,1,3,3-tetramethyldisiloxane. Hexapropyldistannoxane (35 g, 0.0684 mol) and 1,1,3,3-tetramethyldisiloxane (31.5 g, 0.2345 mol) were placed in a round-bottom flask equipped with a reflux condenser, the mixture was heated for 24 h at 70 °C and distilled *in vacuo* to give 32.8 g (94 %) of tripropylstannane, b.p. 50 °C (2 Torr) (*cf.* Ref. 4: b.p. 80—81 °C).

Reaction of dibutyltin oxide with 1,1,3,3-tetramethyldisiloxane. Dibutyltin oxide (11 g, 0.05 mol) and 1,1,3,3-tetramethyldisiloxane (47.4 g, 0.2 mol) were placed in a round-bottom flask equipped with a reflux condenser. After boiling the reaction mixture for 24 h the starting compounds were recovered unchanged.

References

1. K. A. Kochetkov, N. M. Zemlyanskii, N. I. Sheverdina, and E. M. Panov, *Metody elementoorganicheskoi khimii. Germanii, olovo, svinets* [*Methods of Organometallic Chemistry. Germanium, Tin, Lead*], Nauka, Moscow, 1968, 468 (in Russian).
2. G. J. V. Vander Kerk, J. G. Notles, and J. G. A. Luijten, *J. Appl. Chem.*, 1957, **7**, 366.
3. W. P. Neuman and H. Niermann, *Ann.*, 1962, **653**, 164.
4. Ryu Ingam, S. Rozenberg, G. Gil'man, and F. Rikens, *Olovoorganicheskie i germaniiorganicheskie soedineniya* [*Organotin and Organogermanium Compounds*], Izd. Inostr. Lit., Moscow, 1962, 123.

Received April 4, 1995

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 10, pp. 2052—2053, October, 1995.

1066-5285/95/4410-1971$12.50