

Preparative synthesis of trialkylstannanes

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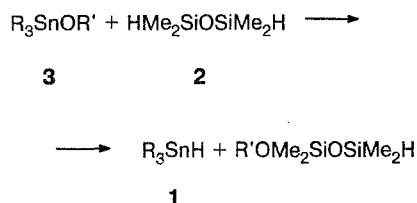
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Reactions of 1,1,3,3-tetramethyldisiloxane with compounds containing an Sn—O bond were studied. Trialkylstannanes are the major products of the reactions with trialkylalkoxy-stannanes and -stannoxanes.

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 tetrahydroaluminate² 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:



R = Me, Bu; R' = Me, R₃Sn

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.

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