Chinese Chemical Letters Vol. 14, No. 4, pp 341 – 342, 2003
http://www.imm.ac.cn/journal/ccl.html

A Novel Access to Organostannane Compounds under Ultrasound Irradiation

Li Zeng PENG, Tian Sheng MEI, Tao ZHANG, Yu Lin LI*

National Laboratory of Applied Organic Chemistry & Institute of Organic Chemistry,
Lanzhou University, Lanzhou 73000

Abstract: A simple and efficient procedure has been developed for the synthesis of organostannane compounds by one-pot reaction of stannane halides, magnesium turnings and organic halides in the presence of 1, 2-dibromoethane under ultrasound irradiation for the first time.

Keywords: Organostannane, one-pot reaction, ultrasound irradiation.

Organostannanes are being found increasing application in organic synthesis[1]. Usually organostannanes are synthesized by the reactions of organolithium or organomagnesium derivatives with trialkyltin halides[2]. Another important method is the radical-induced or Pd-promoted addition of tin hydrides to unsaturated systems (*e.g.*, alkynes, alkenes)[3]. Concerning our interest in the synthesis and chemistry of organostannanes, we report here a new method for the synthesis of organostannanes compounds by one-pot reaction of stannane halides, magnesium turnings and organic halides in the presence of 1, 2-dibromoethane under ultrasonic conditions[4] (**Scheme 1**).

Scheme 1

$$\text{R-X} + \text{R'}_3\text{SnCl} \xrightarrow[\text{THF, sonication, r.t.}]{\text{Mg, BrCH}_2\text{CH}_2\text{Br}} \text{R'}_3\text{Sn-R}$$

R = aryl, allyl, alkyl, vinyl R'=Bu, Me; X=Cl, Br, I

A wide range of stannane compounds was subjected to this procedure to produce the corresponding products in quite high yield. Gram-scale reactions with 0.1-10.0 g of stannane halide were also carried out and found to give analogously good yields of the corresponding products. The results are presented in **Table 1**.

As seen, this methodology can accommodate a variety of organic functional halides, the yields are nearly quantitative. It should be noted that (a) the quality of the reagents

*E-mail: liyl@lzu.edu.cn

is not critical (solvent or halides) and they do not need to be purified before use; and (b) the use of 1, 2-dibromoethane is not necessary but usually it can increase the yield.

Table 1 Improved preparation of organostannane compounds from organic halides

Entry	Organic halide	Organostannane	Yield, %
1	C_6H_5Br	$C_6H_5SnBu_3$	90
2	CH_2=$CHCH_2Br$	CH_2=$CHCH_2SnBu_3$	95
3	$PhCH_2Br$	$PhCH_2SnBu_3$	95
4	$PhCH(CH_3)I$	$PhCH(CH_3)$ $SnBu_3$	95
5	Ph_2CHBr	$Ph_2CHSnBu_3$	95
6	*o*-$C_6H_4(CH_2)_2$	*o*-$C_6H_4(CH_2SnBu_3)_2$	95
7	CH_2=$CHBr$	CH_2=$CHSnBu_3$	90
8	*n*-BuBr	*n*-$BuSnBu_3$	95
9	*s*-BuBr	*s*-$BuSnBu_3$	95

Acknowledgments

This work was financial supported by the National Natural Science Foundation of China (Grant No. 20072012) and the Special Research Grant for Doctoral Sites in Chinese Universities (Grant No. 20010730001).

References and Notes

1. For reviews see: (a) J. K. Stille, *Angew Chem., Int. Ed. Engl.,* **1986**, *25*, 508; (b) V. Farina, V. Krishnamurthy, W. J. Scott, *Org. React*., **1997**, *50*, 1; (c) M. A. J. Duncton, G. Pattenden, *J. Chem. Soc., Perkin Trans. 1*, **1999**, 1235, and references cited therein.
2. (a) S. Thayumanavan, S. Lee, C. Liu, P. BeaK, *J. Am. Chem. Soc.*, **1994**, *116*, 9755; (b) E. Hupe, P. Knochel, *Org. Lett.,* **2001**, *3*, 127.
3. J. P. Ferezou, M. Julia, Y. Li, L. W. Liu, A. Pancrazi, *Synlett.,* **1991**, 53.
4. F. David-Quillot, S. Lunot, D. Marsacq, A. Duchene, *Tetra. Lett.,* **2000**, *41*, 4905 and references cited therein.
5. B. P. Bandgar, V. S. Sadavarte, L. S. Vppalla, *Tetra. Lett.,* **2001**, *42*, 951.
6. The following procedure is representative: A flask containing 10 mL of THF, 480 mg (20 mmol) of magnesium turnings, 0.1 mL of 1, 2-dibromoethane, 3.25 g (10 mmol) of tributy stannane chloide and 2.32 g (10 mmol) of α-methylbenzyl iodide[5] (entry **4**) is plunged into a commercial ultrasonic cleaning bath (KQ-250, working frequency: 40 KHz) and sonicated for 0.5 h. The mixture was treated by the usual way to give 3.75 g (95% yield) of (α-methylbenzyl)tributyltin. IR (neat): 2957 (s), 2928 (s), 2871 (s), 2856 (s), 1600 (m), 1492 (m), 1457 (m), 1376 (m) cm^{-1}; ^1H NMR ($CDCl_3$, 400M Hz, δ ppm) 7.23 (t, 2 H, J = 7.6 Hz), 7.04 (m, 3 H), 2.73 (q, 1 H, J = 7.6 Hz), 1.60 (d, 3 H, J = 7.6 Hz), 1.44-1.24 (m, 15 H), 0.91-0.78 (m, 12 H); EIMS *m/z*: 396 (M^+, 2), 339 (15), 291(81), 179 (100). HRMS (ESI) calcd. for $C_{20}H_{36}Sn$ 396.1840, found 396.1851.

Received 7 June, 2002