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SIMPLE METHOD FOR THE SYNTHESIS OF STANNOLE DIANION

Masaichi Saito^a; Ryuta Haga^a; Michikazu Yoshioka^a Saitama University, Saitama, Japan

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SIMPLE METHOD FOR THE SYNTHESIS OF STANNOLE DIANION

Masaichi Saito, Ryuta Haga, and Michikazu Yoshioka Saitama University, Saitama, Japan

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Reduction of 1-t-butyl-1,2,3,4,5-pentaphenylstannole by lithium gave the stannole dianion. Reduction of the bi(1,1-stannole) having a t-butyl group on the tin by lithium also gave the stannole dianion, the formation of which was evidenced by spectral analysis and chemical trapping reaction.

Keywords: 1-t-Butyl-1-phenylstannole; bi(1,1-stannole); reduction; stannole dianion

Recently, much attention has been focused on anions and dianions of siloles and germoles, heavier congeners of cyclopentadienyl anion having aromaticity. In contrast to the well-investigated dianions of siloles and germoles, no report on dianions of stannole had been published before we succeeded in the first synthesis of a stannole dianion by the reduction of a newly synthesized bi(1,1-stannole) having a phenyl group on the tin. Very recently, we have reported that the reduction of hexaphenylstannole 1 also gave the stannole dianion 2 together with phenyl lithium, as evidenced by H, C, and He Sn NMR analysis. Reductive elimination of a phenyl group on the tin prompted us to investigate the reduction of other stannoles having an alkyl group on the tin. We report herein the synthesis of stannole dianion 2 from t-butyl-substituted stannoles, 3 and 4.

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Address correspondence to Masaichi Saito, Department of Chemistry, Faculty of Science, Saitama University, Shimo-okubo, Sakura-ku, Saitama-city, Saitama 338-8570 Japan. E-mail: masaichi@chem.saitama-u.ac.jp

RESULTS AND DISCUSSION

Reaction of 1-t-butyl-1,2,3,4,5-phenylstannole (3) with lithium in ether gave a deep red solution, suggesting the formation of an anionic species. Treatment of the reaction mixture with methyl iodide gave the 1,1-dimethylstannole $\bf 5$ in 67% yield. The formation of $\bf 5$ was reasonably interpreted in term of the formation of stannole dianion $\bf 2$. Reductive elimination of a t-butyl group on the tin prompted us to investigate the reduction of bi(1,1-stannole) $\bf 4$. Reaction of $\bf 4$ with lithium in THF* also gave a deep red solution. The 13 C NMR spectrum of the reaction mixture showed the formation of stannole dianion $\bf 2^2$ as a main product. Treatment of the resulting solution with methyl iodide gave $\bf 5$ in 21% yield.

In summary, reductive elimination of a *t*-butyl group of stannoles **3** and **4** gave the stannole dianion **2**.

^{*}Bi(1,1-stannole) **5** is sparingly soluble to ether.

REFERENCES

- a) E. Colomer, R. J. P. Corriu, and M. Lheureux, Chem. Rev., 90, 265 (1990);
 b) J. Dubac, C. Gu Rin, and P. Meunier, in The Chemistry of Organic Silicon Compounds, edited by Z. Rappoport and Y. Apeloig (John Wiley and Sons, Chichester, 1998), p. 1961.
- [2] M. Saito, R. Haga, and M. Yoshioka, Chem. Commun., 1002 (2002).
- [3] M. Saito, R. Haga, and M. Yoshioka, Chem. Lett., in press.
- [4] M. Saito, R. Haga, and M. Yoshioka, manuscript in preparation.