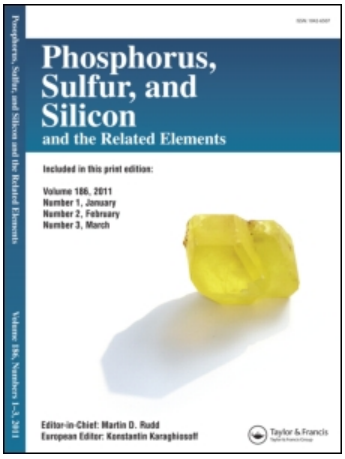


Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

Synthesis of 9,10-Dihydro-9,10-Distannaanthracene

Masaichi Saito; Minehiko Nitta; Michikazu Yoshioka

To cite this Article Saito, Masaichi , Nitta, Minehiko and Yoshioka, Michikazu(2001) 'Synthesis of 9,10-Dihydro-9,10-Distannaanthracene', Phosphorus, Sulfur, and Silicon and the Related Elements, 168: 1, 249 — 251
To link to this Article: DOI: 10.1080/10426500108546562
URL: http://dx.doi.org/10.1080/10426500108546562

Phosphorus Sulfur and Silicon, 2001, Vol. 168, pp. 249-251
Reprints available directly from the publisher
Photocopying permitted by license only

Synthesis of 9,10-Dihydro-9,10-Distannaanthracene

MASAICHI SAITO, MINEHIKO NITTA and MICHIKAZU YOSHIOKA

Department of Chemistry, Faculty of Science, Saitama University, Urawa, Shimo-okubo, Saitama, 338–8570 JAPAN

The 9,9,10,10-tetramethyl-9,10-dihydro-9,10-distannaanthracene has been first synthesized and characterized. The X-ray crystallographic analysis of 9,9,10,10-tetramethyl-9,10-dihydro-9,10-distannaanthracene showed that the central tricyclic framework had a butterfly conformation with the dihedral angle of 143°.

Keywords: 9,10-dihydro-9,10-distannaanthracene; X-ray crystallographic analysis; butterfly conformation

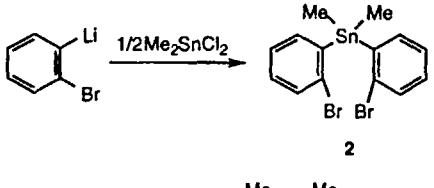
INTRODUCTION

Since the first synthesis of 9,9,10,10-tetraphenyl-9,10-dihydro-9,10-disilaanthracene,[1] studies on the synthesis, structures, and reactions of 9,10-dihydro-9,10-disilaanthracene have been developed.[2] The corresponding germanium analogs, 9,10-dihydro-9,10-digermaanthracenes, were also synthesized and characterized.[3] One of the most interesting features of the dimetalaanthracene derivatives is their synthetic utilities as potential precursors for metal-containing reactive intermediates. Very recently, fascinating silicon-containing reactive species such as a bis(silyl anion)[4] and a relatively stable silyl radical[5] have been reported to be derived from the 9,10-dihydro-9,10-

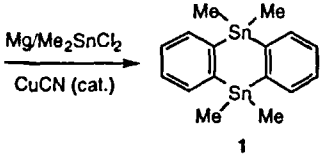
disilaanthracene. As for tin analogs, only one report on the 9,10-dihydro-9,10-distannaanthracene where all aromatic hydrogens have been replaced by fluorine has been found without synthetic and structural details,[6] although there are a few reports on 9,10-dihydroanthracenes having a tin atom at the 10-position.[7], [8] We report herein the first synthesis and structure of 9,9,10,10-tetramethyl-9,10-dihydro-9,10-distannaanthracene (**1**).

RESULTS AND DISCUSSION

(2-Bromophenyl)lithium (2 equiv) prepared by 1,2-dibromobenzene with butyllithium at −100 °C was coupled with dimethyldichlorostannane to give bis(2-bromophenyl)-dimethylstannane (**2**). Reaction of **2** with magnesium and dimethyldichlorostannane in the presence of a catalytic amount of cuprous cyanide afforded 9,9,10,10-tetramethyl-9,10-dihydro-9,10-distannaanthracene (**1**) in a moderate yield, the structure of which was established by X-ray crystallographic analysis (Fig 1). The central six-membered ring has a boat comformation and hence the tricyclic framework has a butterfly conformation with the dihedral angle of 143 °, which is the smallest angle among the dihydrodimetalaanthracene derivatives of group 14 metals.[9]



2



1

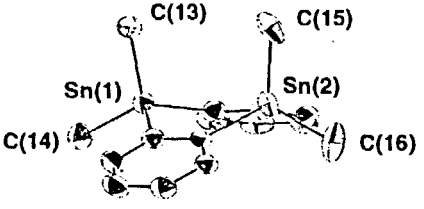


Fig 1. *ORTEP* drawing of **1** with thermal ellipsoids plots (40% probability for non-hydrogen atoms). Selected bond lengths (Å) and angles (deg): Sn(1)–C(13), 2.139(10); Sn(1)–C(14), 2.165(11); Sn(2)–C(15), 2.142(13); Sn(2)–C(16), 2.108(15); C(13)–Sn(1)–C(14), 110.4(4); C(15)–Sn(2)–C(16), 110.5(7).

In ^{1}H NMR spectrum of **1**, there appeared a singlet at δ 0.47 assignable to methyl hydrogens which did not change significantly in the temperature range from 20 to –70 °C, suggesting a rapid boat to boat inversion. It is reasonably assumed that a rapid boat-to-boat inversion occurs in solution as in the case of other tetramethyl derivatives.[8]

Acknowledgment. This work was partially supported by a Grant-in Aid for Encouragement of Young Scientists (No. 10740288) from the Ministry of Education, Science, Sports, and Culture, Japan.

References

[1] (a) H. Gilman and E. A. Zuech, *J. Am. Chem. Soc.*, **82**, 3605 (1960). (b) H. Gilman, E.A. Zuech, and W. Steudel, *J. Org. Chem.*, **27**, 1836 (1962).
[2] For a review, see: T. J. Barton, *Comprehensive Organometallic Chemistry*; 1st Ed., Pergamon Press, Elmsford, NY, **2**, 273 (1982).
[3] O. A. D'yachenko, S. V. Soboleva, and L. O. Atovmyan, *Zh. Strukt. Khim.*, **17**, 496 (1976).
[4] (a) W. Ando, K. Hatano, and R. Urisaka, *Organometallics*, **14**, 3625 (1995). (b) K. Hatano, K. Morihashi, O. Kikuchi, and W. Ando, *Chem. Lett.*, 293 (1997).
[5] S. Kyushin, T. Shinnai, T. Kubota, and H. Matsumoto, *Organometallics*, **16**, 3800 (1997).
[6] G. F. Lanthier, J. M. Miller, S. C. Cohen, and A. G. Massey, *Org. Mass Spectrom.*, 235 (1974).
[7] P. Jutzi, *Chem. Ber.*, **104**, 1455 (1971).
[8] W. Z. McCarthy, J. Y. Corey, and E. R. Corey, *Organometallics*, **3**, 255 (1984).
[9] References cited in ref. 8.