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SYNTHESIS AND CHARACTERISATION OF THE FIRST EXAMPLE OF A TETRASTANNACYCLOHEXANE

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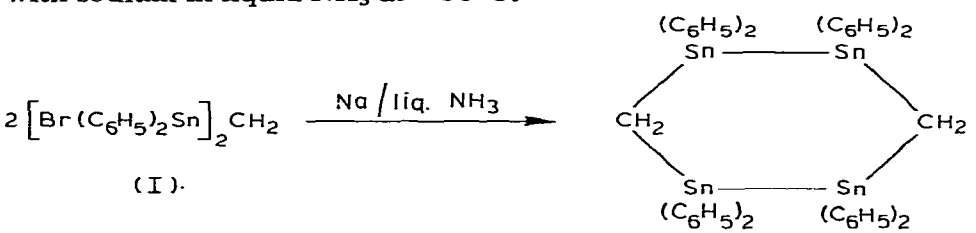
Summary

The synthesis and spectral properties of 2,2,3,3,5,5,6,6-octaphenyl-2,3,5,6-tetrastannacyclohexane are described.

Introduction and results

A recent paper by Kuivila [1] describing the unusual behavior of bis(stannyl)-methanes prompts us to report the preparation of the first example of a tetra-stannacyclohexane starting from an analogous bis(stannyl)methane. We find that bis(bromodiphenylstannyl)methane (I) [2] is readily transformed into 2,2,3,3,5,5,6,6-octaphenyl-2,3,5,6-tetrastannacyclohexane (II) upon treatment with sodium in liquid NH_3 at —80°C.



(I)

(II, 63 %)

Experimental

Compound (II) was obtained by treatment of I with an excess of sodium in liquid NH_3 at —80°C. The product was recrystallized from CH_2Cl_2/CH_3OH,

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(m.p. 180—182°C), it shows in its ^1H NMR spectrum ($CDCl_3$) the expected CH_2 signal at 1.14 ppm ($^2J(^{119}Sn—C—^1H)$ 56.8 Hz; $^3J(^{119}Sn—Sn—C—^1H)$ 22.8 Hz). Its ^{13}C NMR spectrum shows for the same CH_2 group a line at —12.53 ppm ($^1J(^{119}Sn—^{13}C)$ 183.2 Hz; $^2J(^{119}Sn—Sn—^{13}C)$ 66.9 Hz). For the phenyl carbons directly linked to the tin, δ 140.72 ppm ($^1J(^{119}Sn—^{13}C)$ 383.3 Hz; $^3J(^{119}Sn—C—Sn—^{13}C)$ 63.2 Hz; $^2J(^{119}Sn—Sn—^{13}C)$ 18.8 Hz). The *ortho*-carbons resonate at δ 136.87 ppm ($^3J(^{119}Sn—C—^{13}C)$ 41.2 Hz; $^2J(^{119}Sn—Sn—C—^{13}C)$ 8.3 Hz). The *meta*-carbons appear at 128.43 ppm ($^3J(^{119}Sn—C—C—^{13}C)$ 47.3 Hz) and the *para* ones, at 128.47 ppm. The 70 eV mass spectrum of compound II shows complex patterns, such as those corresponding to $(C_6H_5)_6Sn_4(CH_2)_2^+$, $(C_6H_5)_4Sn_3(CH_2)_2^+$ and $(C_6H_5)_3Sn_2CH_2^+$.

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