A Direct Route to a Novel Tetraphenyldisilabicyclo[2.2.2.]octa-2,5-diene: A Tetraphenyldisilene Precursor

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Summary Synthesis of 2:3,5:6-dibenzo-7,7,8,8-tetraphenyl-7,8-disilabicyclo[2.2.2.]octa-2,5-diene from dichlorodiphenylsilane, anthracene and magnesium-magnesium bromide in THF is described; the disilabicyclo compound was found to undergo a retrodiene reaction, affording anthracene and presumably tetraphenyldisilene.

PEDDLE et al. have reported an approach to tetramethyldisilene (Me₂Si=SiMe₂) via thermolysis of the 7,8-disilabicyclo[2.2.2.]2,5-octadiene (1).¹

(1)
$$R = Me$$
(2) $R = Ph$

Heat

 $R_2Si = SiR_2$
 $R_2Si = SiR_2$

We now report the preparation of 2:3,5:6-dibenzo-7,7,8,8-tetraphenyl-7,8-disilabicyclo[2.2.2.]octa-2,5-diene (2) and its thermal decomposition to anthracene and presumably tetraphenyldisilene.

Recently we have reported the formation of octaphenylcyclotetrasilane (3) from a reaction between dichlorodiphenylsilane, magnesium bromide and magnesium metal in tetrahydrofuran (THF).²

$$Ph_2SiCl_2 + Mg - MgBr_2 \xrightarrow{THF} Ph_2Si]_4 + MgCl_2$$
(3)

$$Ph_2SiCl_2 + Mg - MgBr_2 \xrightarrow{THF} (2)$$

(2)
$$\xrightarrow{>320 \text{ °C}} n \left[\text{Ph}_2 \text{Si} = \text{SiPh}_2 \right] \longrightarrow \left[\text{Ph}_2 \text{Si} - \text{SiPh}_2 \right]_n$$

When the reaction² was conducted in the presence of a stoicheiometric amount of anthracene, a white amorphous solid precipitated which was collected by filtration subsequent to hydrolysis of the red-coloured reaction mixture. Washing of this solid material several times with hot benzene followed by addition of hot petroleum ether (b.p. 30-60 °C) to the combined benzene extracts gave a white crystalline compound (2), m.p. 302—306 °C (with slight decomposition) in ca. 40% yield.

The ¹H n.m.r. spectrum [δ 4.62 (2H, s) and 6.72—7.32

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(28H, m)] and the parent peak (m/e 542) were consistent with the structure of (2).†

Thermolysis of (2) above 325° gave an 80% yield of anthracene and a yellow-orange coloured non-volatile solid, presumably formed by polymerization of an incipient tetraphenyldisilene.

A major fragment (m/e 364) which is consistent with the

mass of tetraphenyldisilene supports its formation from (2). We thank the National Science Foundation for support,

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† Satisfactory elemental analyses have been obtained.

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