

NOTE

2,2,7,7-TETRAPHENYL-2,7-DISILAOCTANE

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(Received April 24th, 1969; in revised form May 22nd, 1969)

2,2,7,7-Tetraphenyl-2,7-disilaoctane has been obtained in 37% yield by treating methyldiphenylchlorosilane with 1,4-dichlorobutane in the presence of magnesium in tetrahydrofuran. None of this product was obtained when (a) the Grignard reagent was formed before addition of the chlorosilane, (b) ether was used in place of tetrahydrofuran, or (c) sodium or lithium was used in place of magnesium.

Boiling hydriodic or hydrobromic acid cleaves the Si-Ph bonds (*cf.* ref. 1) to give polymeric material, but no reaction occurs with boiling hydrochloric acid, glacial acetic acid, or acetic anhydride.

EXPERIMENTAL

2,2,7,7-Tetraphenyl-2,7-disilaoctane

A mixture of 1,4-dichlorobutane (6.4 g, 0.05 mole) (or the corresponding amount of 1,4-dibromobutane), methyldiphenylchlorosilane (23.3 g, 0.10 mole), magnesium (2.4 g, 0.1 g-atom), and tetrahydrofuran (100 ml) was stirred for 10 h at room temperature. Hydrochloric acid (10 ml of 5% acid) was added to destroy the unreacted magnesium and dissolve the magnesium salts, the tetrahydrofuran layer was separated, and the solvent was evaporated off to leave a viscous liquid from which crystals separated after 3 weeks at room temperature. The compound was recrystallised by dissolving it in acetone and adding 1.5 volumes of ethanol. Crystals (8.3 g, 37%), m.p. 54°, were obtained. (Found: C, 79.6; H, 7.2; Si, 12.7; mol.wt., 454. $C_{30}H_{34}Si_2$ calcd.: C, 79.7; H, 7.6; Si, 12.5%; mol.wt., 451.) They were insoluble in water and cold ethanol, but soluble in acetone, benzene, carbon tetrachloride, chloroform, toluene, ether, n-hexane, and cyclohexane. The IR spectrum showed the characteristic bands of CH_3-Si at 8 μ , of C_6H_5-Si at 7 μ , 9 μ , and 10 μ , strong bands of $-(CH_2)_4-$ at 6.8 μ and 13.8 μ , and bands of monosubstituted benzene at 13.5 μ and 14.2 μ . The PMR spectrum showed peaks at τ 9.31 (Me), τ 2.54 and 2.3 (Ph), and τ 8.38 and 8.56 $[-(CH_2)_4-]$. The form was as expected for an A_2B_2 system.

Reactions with acids

2,2,7,7-Tetraphenyl-2,7-disilaoctane (0.2 g) was refluxed with 3 ml of concentrated hydriodic acid or hydrobromic acid for 5 h. The reaction mixture was cooled and treated with 10 ml of water and 10 ml of ether. An insoluble solid (*ca.* 0.1 g) was

obtained (rather more with hydriodic than with hydrobromic acid) which did not melt at 400°, and is thought to be highly polymeric.

The starting material was recovered unchanged from similar treatment with hydrochloric acid and from boiling for 2.5 h with acetic acid or acetic anhydride.

ACKNOWLEDGEMENT

This work was supported by a grant from the Chemistry Research Center, National Taiwan University (Grant No. CRC-5609) to which the authors wish to express their thanks.

REFERENCE

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