Journal of Organometallic Chemistry
Elsevier Sequoia S.A., Lausanne
Printed in The Netherlands

NOTE

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

SHENG-LIEH LIU AND HSIOU-CHI CHEN
National Taiwan University, Taipei, Taiwan (Free China)

(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 7μ, 9μ, and 10μ, strong bands of $-(CH_2)_4-$ at 6.8μ and 13.8μ, and bands of monosubstituted benzene at 13.5μ and 14.2μ. 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

1 S. L. Liu and W. F. Chen, *J. Chinese Chem. Soc. (Taiwan) Ser. II*, 13 (1966) 118.