Tetrahedron Letters No. 23, pp. 5-7, 1960. Pergamon Press Ltd. Printed in Great Britain

THE FORMATION OF DODECAPHENYLCYCLOHEXASILANE FROM THE REACTION OF DICHLORODIPHENYLSILANE WITH SODIUM AND WITH LITHIUM

H. Gilman, D. J. Peterson, A. W. Jarvie and H. J. S. Winkler
Chemistry Department, Iowa State University,
Ames, Iowa

(Received 19 September 1960)

FROM the reaction of dichlorodiphenylsilane with sodium, Kipping and co-workers[1] obtained by careful fractionation of the very complex mixture of products a substance called Compound (B). They designated this compound as octaphenylcyclotetrasilane. It was recently reported[2] that their Compound (A) was octaphenylcyclotetrasilane and not a compound containing "two tervalent silicon atoms". There was a high degree of reasonableness at that time for such a structure in view of some uncommon reactions. We have now found that their Compound (B) is dodecaphenylcyclohexasilane. We prepared Compound (B) by the action of sodium or lithium on dichlorodiphenylsilane and showed this compound to be identical with a sample of the original compound prepared many years ago.

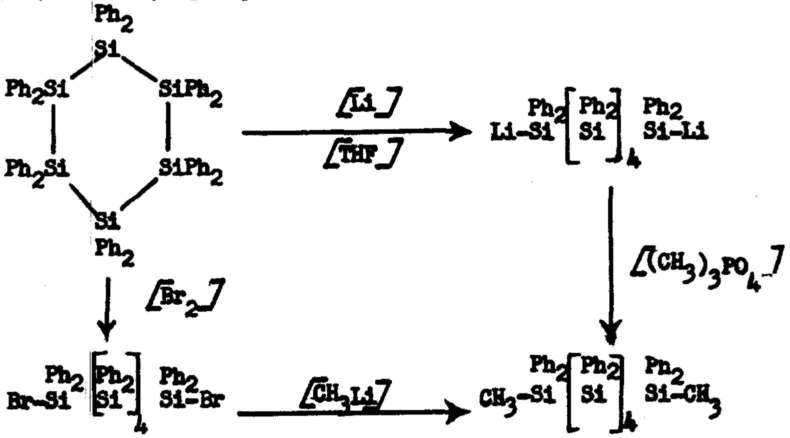
The structure of Compound (B) was established by two different types

---

[1] F.S. Kipping and J.E. Sands, J. Chem. Soc. **119**, 830, 848 (1921); F.S. Kipping, Ibid. **123**, 2590 and 2598 (1923); F.S. Kipping, Ibid. **125**, 2291 (1924); F.S. Kipping, Ibid. 2719 and 2728 (1927); F.S. Kipping and H.E. Murray, Ibid. 360 (1929); A.R. Steele and F.S. Kipping, Ibid. 2545 (1929); F.S. Kipping, Proc. Roy. Soc. A **159**, 139 (1937).

[2] H. Gilman, D.J. Peterson, A.W. Jarvie and H.J.S. Winkler, J. Amer. Chem. Soc. **82**, 2076 (1960).

of cleavage reactions. One of these involved cleavage by lithium metal in tetrahydrofuran to give 1,6-dilithiododecaphenylhexasilane. This dilithium compound was characterized by a quantitative determination of its silyllithium content and by its conversion to 1,6-dimethyldodecaphenylhexasilane (m.p. 183-184°) by trimethyl phosphate.



This compound was shown to be identical with the product obtained by first cleaving Compound (B) by one mole equivalent of bromine in benzene to give 1,6-dibromododecaphenylhexasilane (m.p. 204-206°), and then reacting this dibromo compound with methyllithium. The dimethyl derivative obtained by these two routes were shown to be identical by the method of mixed melting points and the superimposability of their infrared spectra.

(Found: C, 78.97, 79.05, 79.14, 79.23; H, 5.75, 5.68, 5.72, 5.81; Si, 14.90, 15.21. Calc. for $CH_3[Si(C_6H_5)_2]_6CH_3$, ($C_{74}H_{66}Si_6$): C, 79.1; H, 5.88; Si, 14.93.) Hydrogen value (moist piperidine). (Found: 102, 96. Calc. 100) Mol. Wt. (in perylene) (Found: 1170, 1110. Calc.: 1123.)

(Found: C, 78.72, 78.91; H, 5.30, 5.45; Si, 15.37, 15.27. Calc. for $[Si(C_6H_5)_2]_6$, ($C_{72}H_{60}Si_6$): C, 79.07; H, 5.53; Si, 15.40.) Hydrogen value. (Found: 125, 124. Calc.: 123.) Mol. Wt.: (in perylene). (Found: 1115, 1070.

Calc.: 1094.) Mol. Wt. (by isothermal distillation). (Found: 1225, 1200, 1025.)

Acknowledgments - The authors are grateful to Dr. F. B. Kipping for supplying us with a sample of Compound (B) which belonged to the original collection of Dr. F. S. Kipping. This research was supported in part by the United States Air Force under contract AF 33(616)-3510 monitored by Materials Laboratory, Directorate of Laboratories, Wright Air Development Center, Wright-Patterson AFB, Ohio.