Journal of Organometallic Chemistry, 235 (1982) 273—276
Elsevier Sequoia S.A., Lausanne — Printed in The Netherlands

THE CRYSTAL STRUCTURE OF TRIMETHYLTRIPHENYLDISILANE

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(Received April 14th, 1982)

Summary

The crystal structure of trimethyltriphenyldisilane is reported. The Si—Si bond length (2.355(1) Å) is shorter than that in perphenylated homocyclic cyclopolysilanes but somewhat longer than Si—Si bonds reported for permethylated homocyclic cyclopolysilanes.

Introduction

Si—Si bond distances reported for various perphenylated and permethylated homocyclic polysilanes (cf. [1] and references therein) are appreciably longer than those in disilane [2], cyclopentasilane [3] or cyclohexasilane [4] as determined by electron diffraction. Since no simple trialkyl/triaryl-disilane structures have been published, no direct comparison with the structure of disilane derivatives can be made. The title compound was therefore prepared and its X-ray crystal structure determination undertaken.

Experimental

1,1,1-Trimethyltriphenyldisilane was prepared by a published method [5] slightly modified. Hexaphenyldisilane dissolved in dimethoxyethane was cleaved by action of lithium and the triphenylsilyllithium formed was given to a solution of trimethylchlorosilane in dimethoxyethane. After filtration, the solvent was removed and the white needles formed were recrystallized from ethanol. Yield: about 70%, m.p. 104°C.

0022-328X/82/0000—0000/$02.75

TABLE 1

ATOMIC PARAMETERS (X 10^4) FOR THE NON-HYDROGEN ATOMS WITH THEIR e.s.d.'S. THE THERMAL PARAMETERS ARE GIVEN OF THE FORM $T = \exp(-2\pi^2 \sum_{ij} a_i^* a_j^* h_i h_j U_{ij})$ WITH U IN Å^2

Atom	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si(1)	1/3	2/3	3406.4(7)	287(1)	287	343(3)	—143	0	0
Si(2)	1/3	2/3	6077.1(8)	450(2)	450	342(3)	—225	0	0
C(1)	5100(1)	7227(1)	2666(1)	419(5)	413(5)	425(7)	214(3)	21(5)	50(5)
C(2)	5329(1)	6686(1)	1355(2)	501(6)	517(6)	512(8)	238(4)	36(6)	—11(6)
C(3)	6634(1)	7086(1)	849(2)	618(6)	666(7)	650(10)	362(4)	175(6)	38(7)
C(4)	7744(1)	8022(2)	1665(2)	473(6)	764(8)	720(10)	299(5)	140(7)	145(8)
C(5)	7554(1)	8591(2)	2941(2)	436(7)	780(10)	720(10)	146(6)	—18(7)	—6(9)
C(6)	6250(1)	8200(1)	3443(2)	484(6)	605(7)	555(8)	188(5)	11(6)	—69(7)
C(7)	3831(2)	8402(2)	6797(2)	1380(10)	875(9)	633(10)	570(8)	—192(10)	—299(8)

Intensity data, structure determination and refinement

Crystal data: $C_{21}H_{24}Si_2$, F wt: 322.60, a 11.313(2), c 8.817(2) Å V 977.3(6) Å^3 (from single crystal diffractometry), $Z = 2$, space group $P\bar{3}$ (No. 147, from structure refinement), d_x 1.130 Mgm^{-3}, μ(Mo-K_α, λ 0.71073 Å) 1.80 cm^{-1}, approximate crystal size: 0.32 × 0.15 × 0.20 mm.

1283 independent reflexions were collected on an Enraf-Nonius computer controlled, four-circle automatic diffractometer with graphite-monochromated Mo-K_α-radiation. 104 reflexions were recorded with zero intensity and after the determination of the structure these were excluded from the final data set. The positions of four silicon atoms were determined by Patterson synthesis in space group $P3$. All the carbon atoms were located in subsequent Fourier maps. The $P3$ structure (4 silicon and 14 carbon atoms together with 16 hydrogen atoms in positions generated from assumed geometry) was refined by anisotropic least-squares to a final $R = 0.034$. The two independent, but chemically equivalent, Si—Si distances were significantly different (2.361(2) and 2.353(1) Å) and the phenyl rings were markedly distorted. Thus space group $P3$ was rejected and the atomic coordinates were transformed to $P\bar{3}$ (the asymmetric unit is 1/3 of the molecule). Refinement was then repeated which resulted in a final conventional $R = 0.032$ ($R_w = 0.045$) for 1138 observed $[F_0 \geqslant 2\sigma(F_0)]$ reflexions. No

TABLE 2

ATOMIC COORDINATES (X 10^3), ISOTROPIC TEMPERATURE FACTORS (Å^2) AND C—H DISTANCES (Å) FOR THE HYDROGEN ATOMS

Atom	x/a	y/b	z/c	B	C—H
H(2)	458(1)	600(1)	82(2)	4.0(3)	0.94(1)
H(3)	676(1)	665(2)	—7(2)	6.7(5)	1.00(2)
H(4)	863(1)	826(1)	135(2)	6.4(4)	0.94(1)
H(5)	830(2)	921(2)	356(2)	7.1(5)	0.95(2)
H(6)	617(1)	864(1)	433(2)	4.6(3)	0.96(2)
H(7a)	386(2)	838(2)	780(3)	10.8(6)	0.89(3)
H(7b)	466(2)	902(3)	650(3)	12.9(8)	0.88(2)
H(7c)	320(2)	863(2)	648(3)	11.3(7)	0.92(2)

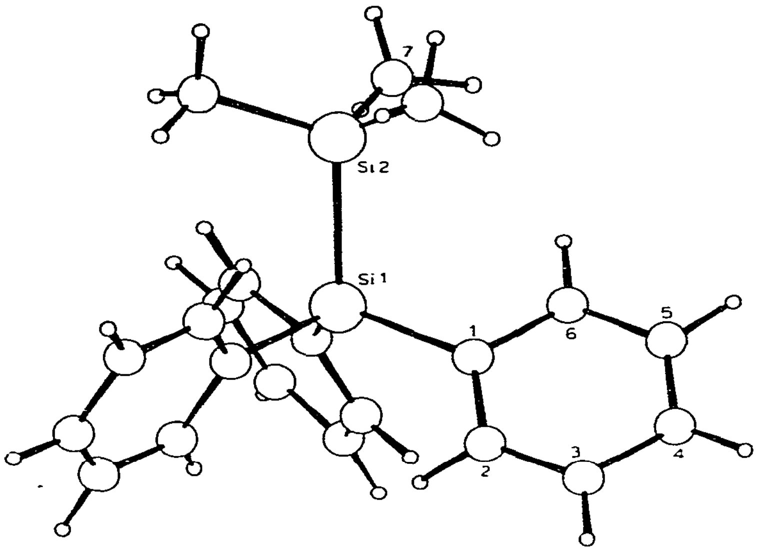

Fig. 1. The diagram of the molecule with the numbering scheme (bare numbers denote carbon atoms).

absorption correction was applied. Final atomic parameters are given in Tables 1 and 2. Tables of observed and calculated structure factors may be obtained from the authors.

Discussion of the structure

The molecular diagram with the numbering scheme is shown in Fig. 1. The bond lengths and angles are given in Table 3. The molecules possessing threefold symmetry are situated on the symmetry axes ±1/3, ±2/3, $\pm z$. The relative position of the Me groups and Ph rings can be characterized by the torsion angle C(Me)—Si—Si—C(Ph) 34.2(1)°. The Si—Si bond length (2.355(1) Å) is shorter than that in perphenylated homocyclic cyclopolysilanes [(Me-t-BuSi)$_4$: 2.377(1) Å [6], (Ph$_2$Si)$_4$: 2.377(1) Å [7], (Ph$_2$Si)$_5$: 2.395(8) Å [8], (Ph$_2$Si)$_6$: 2.394(3) Å [1] but somewhat longer than Si—Si bonds reported for permethyl-

TABLE 3

BOND LENGTHS (Å) AND BOND ANGLES (°) (with e.s.d.'s)

Si(1)—Si(2)	2.355(1)	C(1)—C(2)	1.392(2)	C(3)—C(4)	1.373(2)
Si(1)—C(1)	1.886(1)	C(1)—C(6)	1.393(2)	C(4)—C(5)	1.366(2)
Si(2)—C(7)	1.862(2)	C(2)—C(3)	1.384(2)	C(5)—C(6)	1.384(2)

Angles			
Si(2)—Si(1)—C(1)	110.3(1)	C(2)—C(1)—C(6)	116.8(2)
C(1)—Si(1)—C(1)′	108.7(1)	C(1)—C(2)—C(3)	121.8(2)
Si(1)—Si(2)—C(7)	109.9(1)	C(2)—C(3)—C(4)	119.9(2)
C(7)—Si(2)—C(7)′	109.0(1)	C(3)—C(4)—C(5)	119.8(2)
Si(1)—C(1)—C(2)	122.6(1)	C(4)—C(5)—C(6)	120.4(2)
Si(1)—C(1)—C(6)	120.6(1)	C(1)—C(6)—C(5)	121.4(2)

ated homocyclic cyclopolysilanes $(Me_2Si)_6$: 2.338(3) Å [9], hexadecamethylbicyclo[3.3.1]nonasilane: 2.341(3) Å [10].

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